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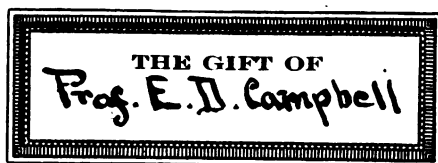
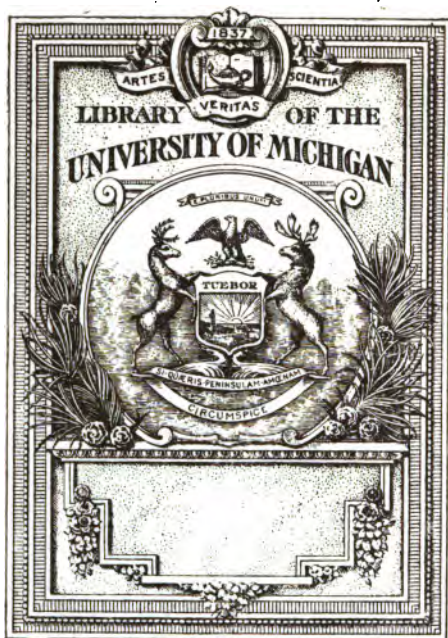
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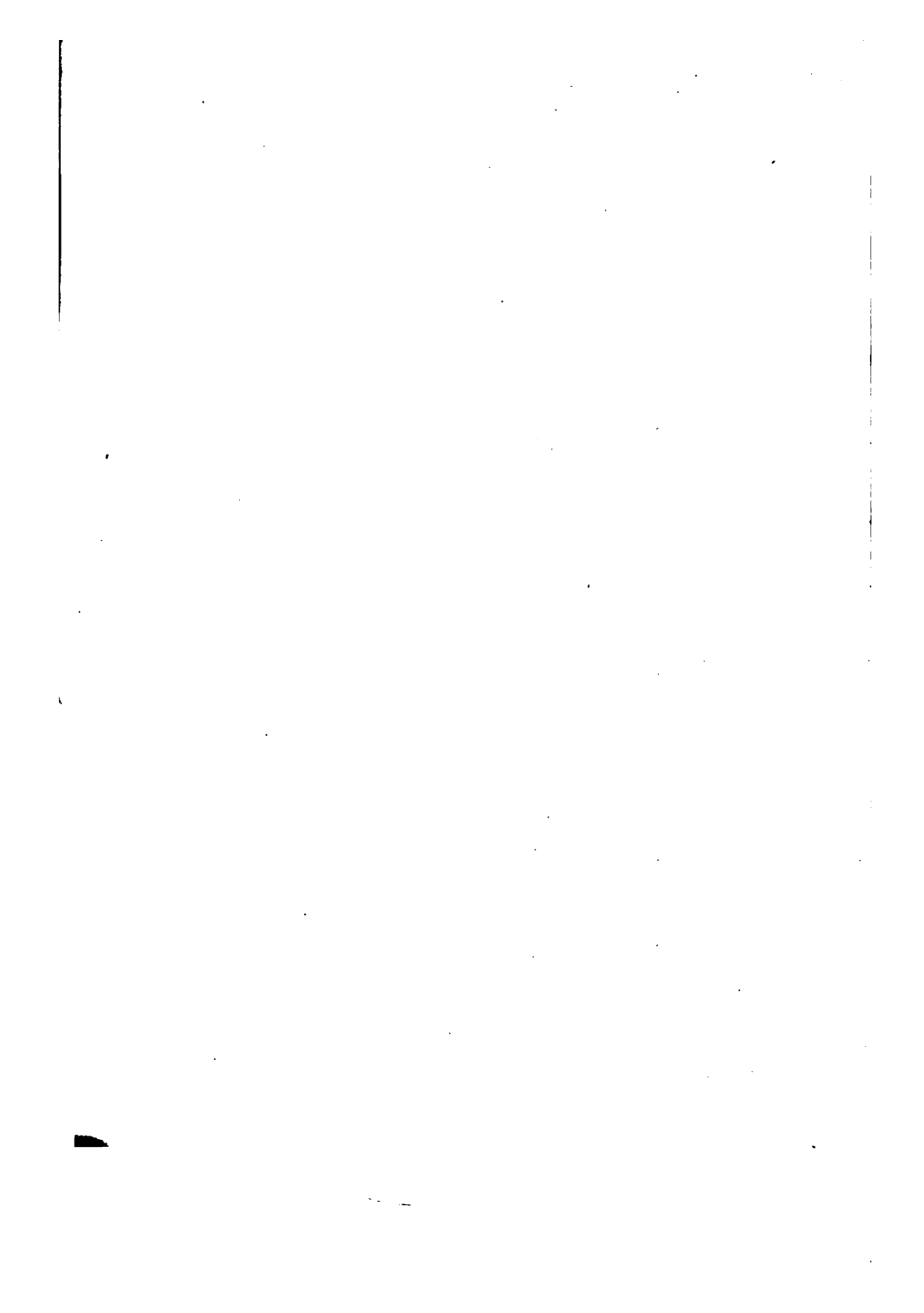
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EXERCISES IN CHEMISTRY

SYSTEMATICALLY ARRANGED TO ACCOMPANY



McPHERSON AND HENDERSON'S

ELEMENTARY STUDY OF CHEMISTRY

BY

WILLIAM MCPHERSON, PH.D.

AND

WILLIAM EDWARDS HENDERSON, PH.D.

GINN & COMPANY

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PREFACE

The exercises contained in this book are systematically arranged to accompany McPherson and Henderson's *An Elementary Study of Chemistry*. The authors believe that the course in chemistry for secondary schools should consist of three recitations and two laboratory periods weekly for one year, and that the laboratory periods should consist of two consecutive recitation hours of at least forty-five minutes each. In accordance with this view an effort has been made to have the exercises of such length that each can be completed in one and one-half hours.

It is no longer necessary to emphasize the importance of laboratory work as a part of the course in elementary chemistry, since it is universally admitted that some laboratory experience is essential for a thorough comprehension of the subject. It is none the less true, however, that laboratory work is of very doubtful value unless carefully directed by an experienced teacher toward some definite end. It is in the hope of aiding such a teacher that this little exercise book has been prepared.

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Campbell, P. H. - 10-12-18 J.

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INFORMATION REGARDING APPARATUS AND CHEMICALS

The lists following include the apparatus and chemicals required for the experiments in this notebook. It is always best to furnish each student with as complete an outfit as possible and to hold him responsible for the same. Certain pieces may, however, be used in common by a number of students and these have been placed in a separate list. It is always cheapest to purchase the apparatus and chemicals in as large quantities as possible. The amounts of most of the chemicals needed for a class of ten are so small that the cost of the same will be proportionately much greater than when larger quantities are ordered. It is always best to order the definite amounts of chemicals listed in the catalogues, such as 100 g. or 1 lb. The supplies may be obtained from any of the large dealers. The following are the addresses of some of the largest firms. Catalogues will be sent on application and should be in every school.

Eimer & Amend, 205 Third Avenue, New York City.

The Bausch & Lomb Optical Company, Rochester, N.Y.

The Arthur H. Thomas Company, Twelfth and Walnut Streets, Philadelphia, Pa.

E. H. Sargent & Co., 145 Lake Street, Chicago, Ill.

The C. H. Stoeltling Company, 45 West Randolph Street, Chicago.

A list of the supplies needed should be sent to a number of firms for quotation on prices. In ordering any piece of apparatus a certain form in some catalogue should be designated; otherwise it will be impossible to compare the prices. In general it is best to purchase as simple a form of apparatus as possible; for example, 20 cents will buy a Bunsen burner which for ordinary purposes is preferable to those costing \$1.00. In the case of large orders a considerable saving may be made by placing the order four or five months early and requesting the dealer to import duty free such articles as can be obtained in this way at a less cost. A much higher price will have to be paid for small orders placed for immediate delivery. A person experienced in the purchase of supplies will always find it possible to materially reduce the cost of the order. The authors will be glad to correspond with teachers in regard to any questions that may arise in the purchase of their supplies or the equipment of laboratories in general.

APPARATUS REQUIRED FOR EACH STUDENT

	APPROXIMATE COST
Beakers, nest of 7, 100 cc. to 700 cc.	\$0.90
Blowpipe	.10
Bottles. Wide mouthed, 1 (60 cc.), 5 (250 cc.)	.10
Bunsen burner	.20
Burner, wing top for bending glass tubing	.10
Calcium chloride drying tube, straight, 15 cm. in length	.10
Clamp, iron burette	.25
Clamp, screw	.10
Clay pipe	.01
Deflagrating spoon	.15
Dish, evaporating, diameter about 7.5 cm.	.20
Dish, hemispherical iron, diameter about 8 cm.	.05
Dish, lead, diameter about 6 cm., 3 cm. in depth	.05
File, round, about 15 cm. in length	.05
File, triangular, about 15 cm. in length	.10
Filters, 25, diameter about 11 cm.	.05
Flasks, 2 (250 cc.), 1 (500 cc.)	.50
Funnel, diameter about 6.5 cm.	.10
Funnel tube, external diameter of tube, 6 mm.	.10
Glass tubing, 100 g., soft, external diameter 6 mm., walls 1 mm. thick	.10
Glass tubing, hard, 1 piece, 30 cm. in length, internal diameter 1 cm.	.06
Glass tubing, hard, 1 piece, 20 cm. in length, internal diameter 6 mm.	.05
Glass rod, 2 pieces, 15 cm. in length, diameter 3 mm.	.05
Mortar, porcelain, diameter about 8 cm.	.40
Porcelain crucible and lid, diameter about 3.5 cm.	.10
Retort, glass stoppered, 150 cc.	.25
Ring stand and rings	.40
Rubber tubing, 1 piece, 75 cm. in length for Bunsen burner, internal diameter 6 mm.	.15
Rubber tubing, 1 piece, 60 cm. in length for connections, etc., internal diameter 5 mm.	.10
Splints, 1 package	.01
Test tubes, 12, 12 cm. in length, diameter about 1.7 cm.	.15
Test tube, graduated, 30 cc.	.25
Test tube, hard glass, preferably Jena glass, 15 cm. in length, diameter about 1.8 cm.	.05
Test tube rack	.20

CHEMICALS REQUIRED FOR A CLASS OF TEN ix

	APPROXIMATE COST
Watch glass, diameter 8 cm.	\$0.05
Window glass, 4 pieces, 10 cm. square	.10
Wire gauze, 2 pieces, 12 cm. square	.10

APPARATUS FOR GENERAL USE FOR A CLASS OF TEN

Balance. Made to carry a load of 100 g. and be sensitive to 1 cg. . .	8.00
Blast lamp, bellows, and rubber tube	6.00
Burettes, 6, capacity 50 cc., graduated to 0.1 cc.	6.00
Condenser (Liebig's), glass, with rubber tubing and large clamp . .	2.00
Corks, best grade, sizes 7, 8, 9, 10, 12, 2 gross	.40
Cork borers, set of 6	.45
Cylinder, graduated, 500 cc.	.50
Cobalt glass, 6 pieces, 10 cm. square	.30
Magnet, small horseshoe	.10
Magnifying glass, small	.40
Pneumatic trough, 6, made of galvanized iron, 30 cm. in length, 20 cm. in depth, 18 cm. in width	2.00
Platinum wire, 3 pieces, small, 8 cm. in length	.75
Weights, 50 g. to 1 cg.	.50

CHEMICALS REQUIRED FOR A CLASS OF TEN

	APPROXIMATE AMOUNT IN GRAMS	APPROXIMATE COST
Acids, Hydrochloric (den. 1.2)	1500	\$0.35
Nitric (den. 1.4)	1000	.20
Pyrogallie	100	.45
Sulphuric (den. 1.84)	2000	.35
Alcohol	100	.10
Alum (ammonium)	25	.01
Aluminium (turnings or filings)	20	.05
Ammonium carbonate	25	.02
Ammonium chloride	250	.05
Ammonium hydroxide (den. 0.90)	1500	.25
Ammonium nitrate	100	.05
Ammonium sulphate	100	.05
Ammonium sulphide solution	250	.10
Antimony	15	.05
Arsenic	2	.01
Arsenic trioxide	10	.02

x CHEMICALS REQUIRED FOR A CLASS OF TEN

	APPROXIMATE AMOUNT IN GRAMS	APPROXIMATE COST
Barium chloride	20	\$0.02
Bismuth	5	.05
Boneblack	100	.01
Borax	75	.05
Boric acid	10	.02
Cadmium	5	.03
Cadmium chloride	10	.04
Calcium chloride (fused or granular)	200	.15
Calcium fluoride (fluorspar)	30	.01
Calcium sulphate	50	.01
Carbon disulphide	30	.01
Charcoal, 10 pieces about 8 cm. x 4 cm.		.20
Cobalt nitrate	10	.05
Copper (turnings or scrap)	250	.20
Copper foil (very thin)	20	.04
Copper oxide (black)	50	.06
Copper sulphate	100	.03
Glucose (sirup)	1000	.10
Gypsum (crystals)	25	.01
Iron chloride (ferrie)	10	.02
Iron powder	25	.01
Iron sulphate (ferrous)	75	.01
Iron sulphide	250	.05
Lead monoxide	25	.01
Lead nitrate	50	.02
Lead peroxide	25	.03
Litmus	10	.02
Litmus paper, 100 strips red and 100 strips blue		.10
Magnesium carbonate	50	.02
Magnesium sulphate	20	.02
Magnesium wire or ribbon	5	.10
Manganese chloride	40	.04
Manganese dioxide	1000	.12
Mercury	20	.05
Mercuric chloride	20	.05
Mercuric oxide	15	.04
Mercurous nitrate	10	.03
Nickel nitrate	10	.02
Paraffin	10	.01
Phenol phthalein	5	.05
Phosphorus	10	.02
Potassium bromide	50	.07

CHEMICALS REQUIRED FOR A CLASS OF TEN xi

	APPROXIMATE AMOUNT IN GRAMS	APPROXIMATE COST
Potassium carbonate	10	\$0.02
Potassium chlorate	250	.15
Potassium chloride	100	.04
Potassium chromate	10	.01
Potassium chromium sulphate	10	.02
Potassium dichromate	125	.08
Potassium hydroxide	50	.03
Potassium iodide	50	.35
Potassium nitrate	25	.02
Potassium permanganate	10	.01
Silver nitrate	10	.13
Sodium	5	.03
Sodium bicarbonate	20	.01
Sodium carbonate	100	.03
Sodium hydrogen phosphate	20	.01
Sodium hydroxide	500	.25
Sodium nitrate	250	.15
Sodium nitrite	75	.07
Sodium silicate (water glass) solution	100	.02
Sodium sulphate crystals	10	.01
Sulphur	250	.02
Tin	35	.04
Zinc (granulated)	1000	.30
Zinc sulphate crystals	100	.03

NOTE. — This list does not include substances always easily obtained, such as sugar, salt, lard, lime, clay, starch, marble, cotton, iron wire, lead (pieces cut from lead pipe).



EXERCISES IN CHEMISTRY

PRELIMINARY EXERCISE

MANIPULATION OF GLASS TUBING

NOTE. — In all operations requiring the application of a strong heat to glass, the heat must be applied gently at first. Highly heated glass must be cooled slowly; otherwise it is easily broken.

1. *Cutting glass tubing.* Place the tubing on the desk and draw the edge of a triangular file across the point at which you wish to cut the glass. After the glass has been scratched, take the tube in the hands with the thumbs placed near together,

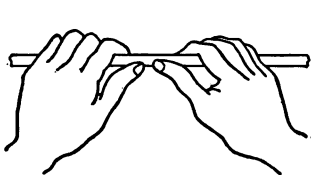


FIG. 1

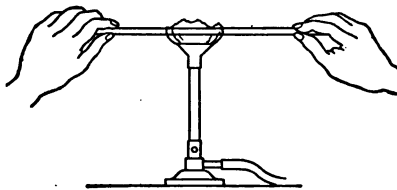


FIG. 2

just back of the scratch (Fig. 1), and gently pull the glass apart, at the same time exerting a slight pressure with the thumbs. If the tube does not yield readily to a gentle pressure, a deeper scratch must be made. In the case of large tubing it may

be found necessary to file a groove around the glass. The edges of the cut tube will be sharp. They should be rounded by rotating them in the tip of the Bunsen flame.

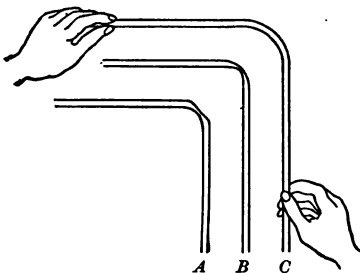


FIG. 3

2. *Bending glass tubing.* Use the ordinary illuminating gas flame, or the luminous Bunsen flame, spread out by

means of the so-called "wingtop" burner. Hold the tube lengthwise in the flame, gently rotating it so that all sides may be equally heated (Fig. 2). Continue the heating until

the glass *easily* bends; then quickly remove it from the flame and bend to the desired shape (Fig. 3, *C*). Great care must be taken to heat the tube uniformly, otherwise the bore of the tube will be contracted (Fig. 3, *A, B*), forming a bend which is not only unsightly but is easily broken.

3. *Inserting glass tubes in corks.* The cork should be of such a size that the smaller end will just enter the flask or bottle in which it is to be used. Soften the cork by means of a press or by rolling it between the desk and a piece of wood. To insert a glass tube select a borer slightly smaller than the tube. Place the cork on the desk and cut through it, not by punching but by rotating the borer under gentle pressure (Fig. 4). Care must be taken to keep the borer at right angles to the cork. The hole should be straight and smooth. The glass tube rounded at the edges is now inserted by a gentle screwlike motion. If the hole is too

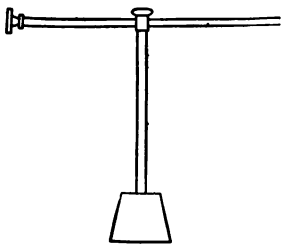


FIG. 4

small to admit the tube when a gentle pressure is applied, it may be slightly enlarged with a round file.

4. *To draw out a glass tube to a small bore.* Heat a portion of the tube in the Bunsen flame until the walls of the heated portion thicken and the size of the bore diminishes (Fig. 5, *A*). The tube must be constantly rotated to prevent the fused portion from sagging. Now quickly

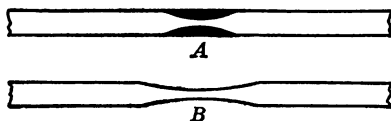
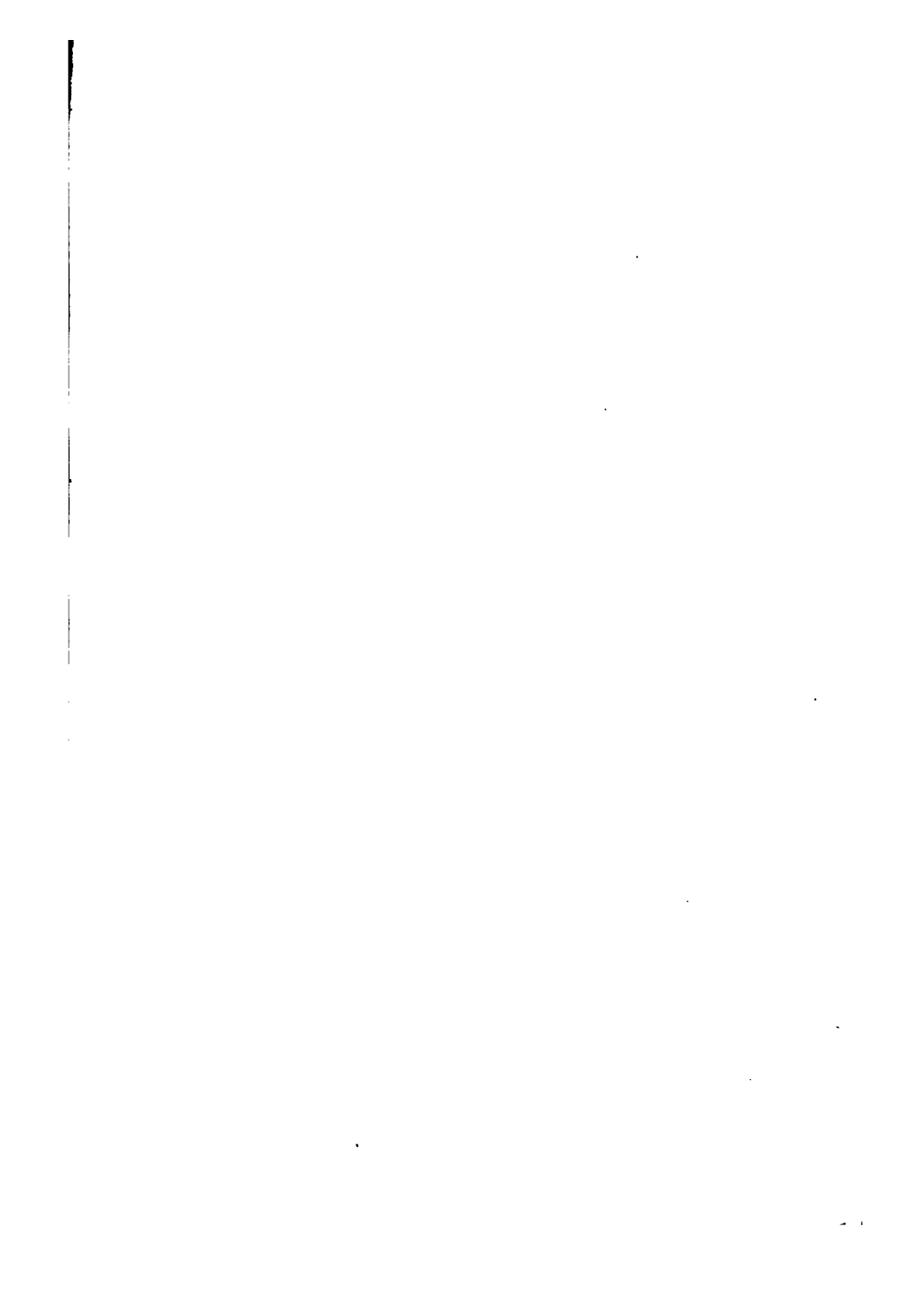


FIG. 5

remove the tube from the flame, and, holding it in a vertical position, gently pull the ends apart until the bore is of the desired size (Fig. 5, *B*). A glass jet may be formed by cutting the tube at *B*, and rounding the edges in a flame.



EXERCISE 1**SOME PRELIMINARY MANIPULATIONS**

1. *Heating liquids in test tubes.* Hold the test tube between the thumb and fingers (Fig. 6), constantly rotating it so as to apply the heat uniformly. The heat should be applied to the upper portion of the liquid, care being taken, however, that the flame does not strike the tube above the level of the

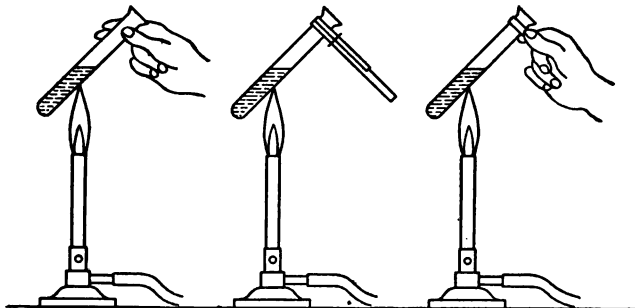


FIG. 6

FIG. 7

FIG. 8

liquid. In case the upper part of the tube becomes heated, it may be supported by a test-tube holder (Fig. 7) or by a band of paper wrapped about the upper part of the tube (Fig. 8). The sudden formation of vapor sometimes causes the contents of the tube to be thrown out; hence care must be taken not to point the tube toward any one.

Half fill a test tube with water and apply heat until the water boils rapidly.

2. *Pouring liquids from one vessel to another.* In doing this care must be taken to prevent the liquid from running down the side of the vessel from which it is poured. A glass rod should be held lightly against the rim of the vessel, as shown in Fig. 11. The liquid will flow down the rod.

Fill a beaker with water and transfer it slowly to another vessel without using the glass rod; repeat, using the glass rod.



EXERCISE 1 (continued)

In pouring liquids from bottles a glass rod may be used; or the neck of the bottle may be placed lightly against the rim of the vessel into which the liquid is being poured (Fig. 10). This will prevent the liquid from running down the side of



FIG. 9

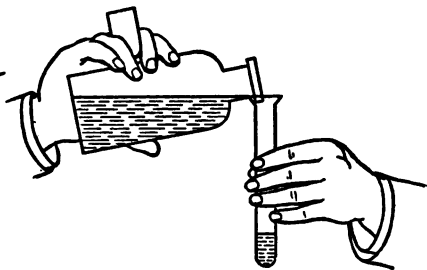


FIG. 10

the bottle. The stopper must never be laid down on the desk. Catch it between the fingers, as shown in Fig. 9. This leaves the hand free to grasp the bottle, as shown in Fig. 10.

3. *Decantation; filtration; solution; evaporation.* It is often necessary to separate a liquid from a finely divided solid which is suspended in it. This may be done by one of the two following methods.

(a) *Decantation.* When the solid is heavy and readily settles to the bottom of the vessel the liquid may be carefully poured off or *decanted*. Thus sand suspended in water may be separated from the liquid in this way.

(b) *Filtration.* Usually, however, the solid will not readily settle or will do so only after long standing. In such cases the mixture is *filtered*, i.e. poured on a filter paper, which allows the liquid to run through but retains the solid. To prepare the filter paper fold it along a diameter into halves, then at right angles to the first fold into quarters. The folded filter is then opened so as to form a cone, half of which is composed of



EXERCISE 1 (continued)

three thicknesses of paper and the remainder of one thickness. Now fit the cone into a funnel of such a size that the paper does not quite reach the top. The paper must accurately fit the funnel; if it does not, make it do so by varying the fold. Place the paper in the funnel and thoroughly wet it with water. After the water has run through

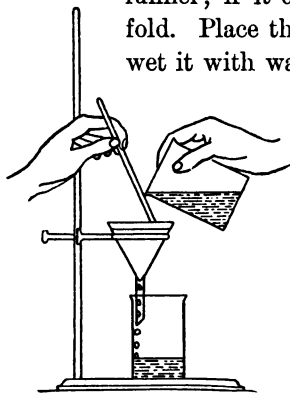


FIG. 11

press the paper firmly against the sides of the funnel with the finger so as to remove any air bubbles. The filter is now ready for use (Fig. 11). The process of filtration not only enables us to separate liquids from solids but also certain solids from each other. To illustrate this, grind a piece of chalk to a powder in a mortar, and mix the product intimately with about an equal bulk of common

salt. Place the mixture in a small beaker, add about 50 cc. of distilled water, and stir with a glass rod. (The ends of the rod should be rounded by rotating them in a flame, otherwise the beaker may be scratched and broken.) The salt dissolves, forming a *solution*. Filter off the insoluble chalk, collecting the *filtrate*, that is, the clear liquid which passes through the filter paper,

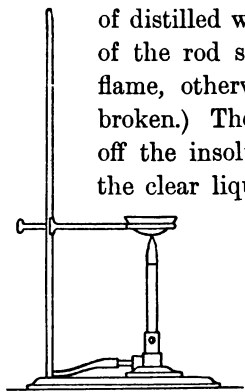
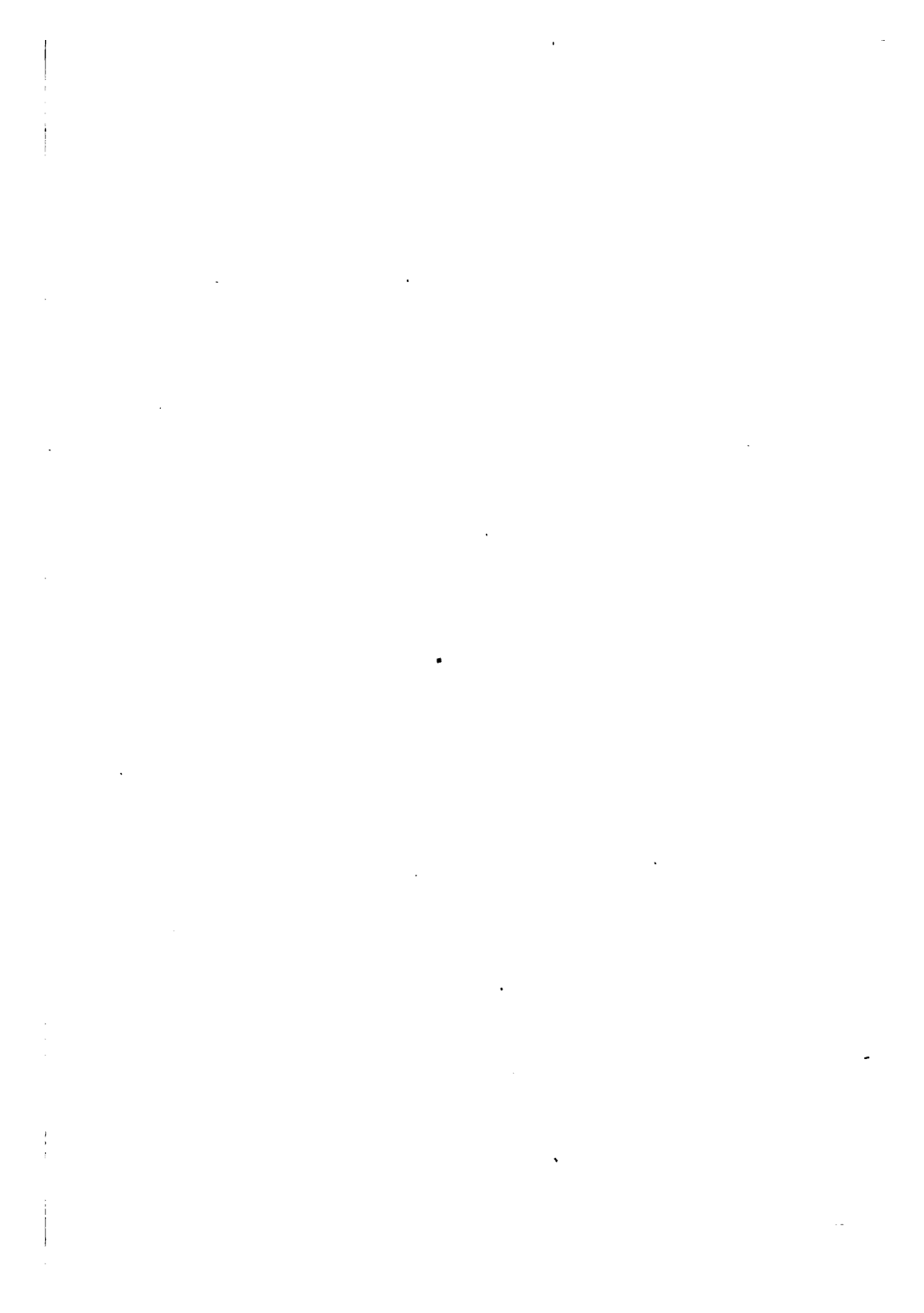


FIG. 12

in a beaker (Fig. 11). The salt may be recovered from the filtrate by the process of *evaporation*. To perform this operation, pour the filtrate into an evaporating dish, support the dish on a ring stand (Fig. 12), and heat gently. The liquid may be made to simmer, but should not actually boil.

Withdraw the heat as soon as the water is evaporated. Note the residue left. Convince yourself that it is common salt.



I. PHYSICAL AND CHEMICAL CHANGES

1. Hold a piece of iron wire in the Bunsen flame for a few seconds. Is the iron changed? Examine it when cooled. What are such changes called?

2. Repeat 1, using a splint of wood in place of the iron wire. How does the change produced differ from that in 1? What are such changes called?

3. Place enough sugar in a clean, dry test tube to cover the bottom to a depth of 1 cm. Heat it gently in the tip of the flame *as long as any changes are produced*. Note all the changes. Is the product sweet? Is it soluble in water? Is the change physical or chemical? Give reason for your answer.

II. ELEMENTS, CHEMICAL COMPOUNDS, MECHANICAL MIXTURES

1. What is an element? Are iron and sulphur included in the list of elements? Weigh out on paper 2 g. of each and study their properties carefully. Try the effect of a magnet on each. Now mix the two, and grind them together intimately in a mortar. Examine the product with a magnifying glass. Can you distinguish the iron from the sulphur? Can you separate them with a magnet? Have they undergone any change in properties? What is such a mixture called?

2. Place the mixture in a clean test tube and heat gently. As soon as the mass begins to glow, quickly withdraw the tube from the flame. Does the glow continue? Heat the tube strongly for a few minutes; then cool and break it, and examine with a magnifying glass the product formed. Can you distinguish between the iron and the sulphur now? Note the effect of a magnet. Of what is the substance composed? When elements combine chemically do they retain their original properties? What is the product of such a combination called?



EXERCISE 3**I. COLLECTION OF GASES**

Fill a wide-mouthed bottle (250 cc.) with water. Cover its mouth with a glass plate, being careful to exclude all air bubbles. Hold the plate firmly in place, invert the bottle, and dip its mouth into the water in a pneumatic trough. Remove the glass plate. Why does the water remain in the bottle? Now fill the bottle with exhaled air by placing one end of a piece of glass or rubber tubing under the mouth of the bottle and blowing gently through the other end.

Before the bottle, so filled, is removed from the trough, cover its mouth tightly with a glass plate. The bottle so covered may then be placed on the desk in either an upright or an inverted position. (When should it be placed in an inverted position?)

Fill a bottle with exhaled air and then transfer the air to another bottle. Draw a diagram to show the method of doing this.

II. PREPARATION OF OXYGEN FROM MERCURIC OXIDE

In the bottom of a clean, dry test tube place about $\frac{1}{2}$ g. of mercuric oxide. This is done by placing the oxide near the end of a narrow strip of folded paper and introducing it carefully into the tube, as shown in Fig. 13. On inclin-



FIG. 13

ing the tube and gently tapping the paper the oxide will be deposited in the bottom of the tube. The paper is now withdrawn, leaving the sides of the tube perfectly clean. Now hold the tube between the thumb and fingers (Fig. 6), and apply a gentle heat to the oxide. The tube must be rotated constantly to distribute the heat, otherwise it may be melted. During the heating insert a glowing splint from time to time into the mouth of the tube. Note the result. Continue to heat as long as any gas is evolved. What remains in the tube? How has the heat affected the mercuric oxide?

EXERCISE 4

I. PREPARATION OF OXYGEN FROM POTASSIUM CHLORATE

Usual laboratory method. Prepare an apparatus according to Fig. 14. *B* is a hard-glass test tube, *D* a glass tube bent at right angles, and *C* a piece of rubber tubing. Mix intimately on paper 6 g. of potassium chlorate and 3 g. of manganese dioxide. The presence of impurities in the materials may lead to a serious explosion when heat is applied; hence test a small portion of the mixture, say $\frac{1}{2}$ g., by heating it in a test tube. In the absence of impurities the oxygen is evolved quietly, unaccompanied by any very marked sparking in the materials.

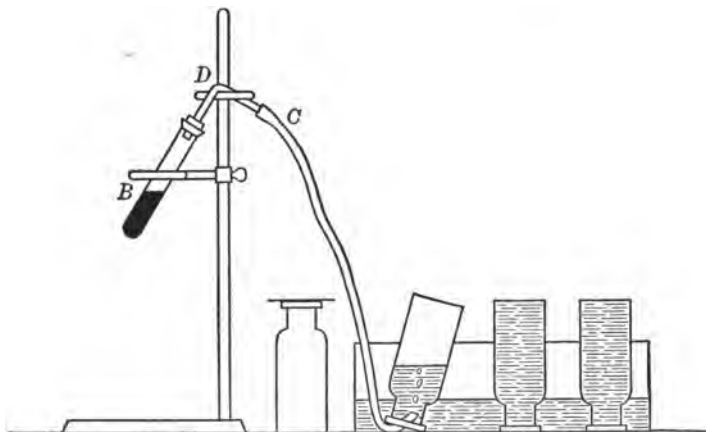


FIG. 14

If pure, transfer the remainder of the mixture to a hard-glass tube, and insert the cork (Fig. 14); then, holding the burner in the hand, heat the mixture gently with a small flame, applying the heat at first to the upper part of the mixture. The flame must not strike the upper part of the test tube, as the cork may be ignited. At first the heat expands the air and a

EXERCISE 4 (continued)

few bubbles escape; then the oxygen is evolved. Regulate the heat so as to secure a uniform and not too rapid evolution of the gas. Collect three or four bottles (250 cc., wide mouthed) of the gas. Before the heat is withdrawn remove the cork from the tube. Why? What is the source of the oxygen? What is the function of the manganese dioxide?

II. PROPERTIES OF OXYGEN

1. Note the physical properties of the gas. (The slight cloud that is often present when oxygen is prepared from potassium chlorate is due to an impurity, and will disappear if the gas is allowed to stand over water.)

2. Repeatedly thrust a glowing splint into a bottle of the gas.

3. Heat some sulphur in a deflagrating spoon until ignited. Note the color and size of the sulphur flame. Now lower the burning sulphur into a bottle of oxygen and note the change.

4. Tip a piece of picture-frame wire (12 cm.—15 cm. long) with sulphur by wrapping a very small bit of cotton about the end of the wire and dipping this into melted sulphur. Ignite the sulphur by holding it in a Bunsen flame for an instant, and then thrust the wire into a bottle of oxygen.

Describe the results obtained in 2, 3, and 4. Are the changes physical or chemical? What becomes of the oxygen?



EXERCISE 5

I. PREPARATION OF HYDROGEN

1. *From water.* Fill a test tube with water and invert it in a beaker of water. Wrap a piece of sodium, the size of a small pea, in a bit of filter paper previously moistened with coal oil. Raise the inverted test tube until the mouth of it dips just below the surface of the water in the beaker and quickly insert the sodium. Stand at arm's length, as a slight explosion sometimes occurs. Notice that the sodium decomposes the water, liberating a gas which is caught in the tube. Test the gas so obtained by quickly inserting a burning splint. Does it act like oxygen? What is the source of the gas? What other methods may be employed for obtaining it from the same source?

2. *From acids* (usual laboratory method). Prepare a hydrogen generator according to Fig. 15. *D* represents a wide-mouthed

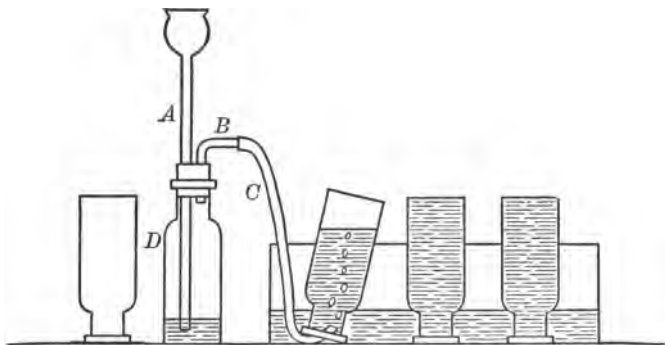


FIG. 15

bottle of about 250 cc. capacity. The gas delivery tube *B*, *C* is the same as that used in the preparation of oxygen (Fig. 14). The funnel tube *A* must extend nearly to the bottom of the bottle. Why? Put 10 g. of granulated zinc (why granulated?)

into *D*, and add a few drops of a solution of copper sulphate. Now pour just enough water through the funnel tube to cover the zinc. Prove that the apparatus is air tight by blowing into the delivery tube until the water is forced nearly to the top of the funnel tube; then quickly close the rubber tube either by tightly pinching it or by placing the tongue firmly against its end. If the apparatus is air tight, the water in the funnel tube will not fall.

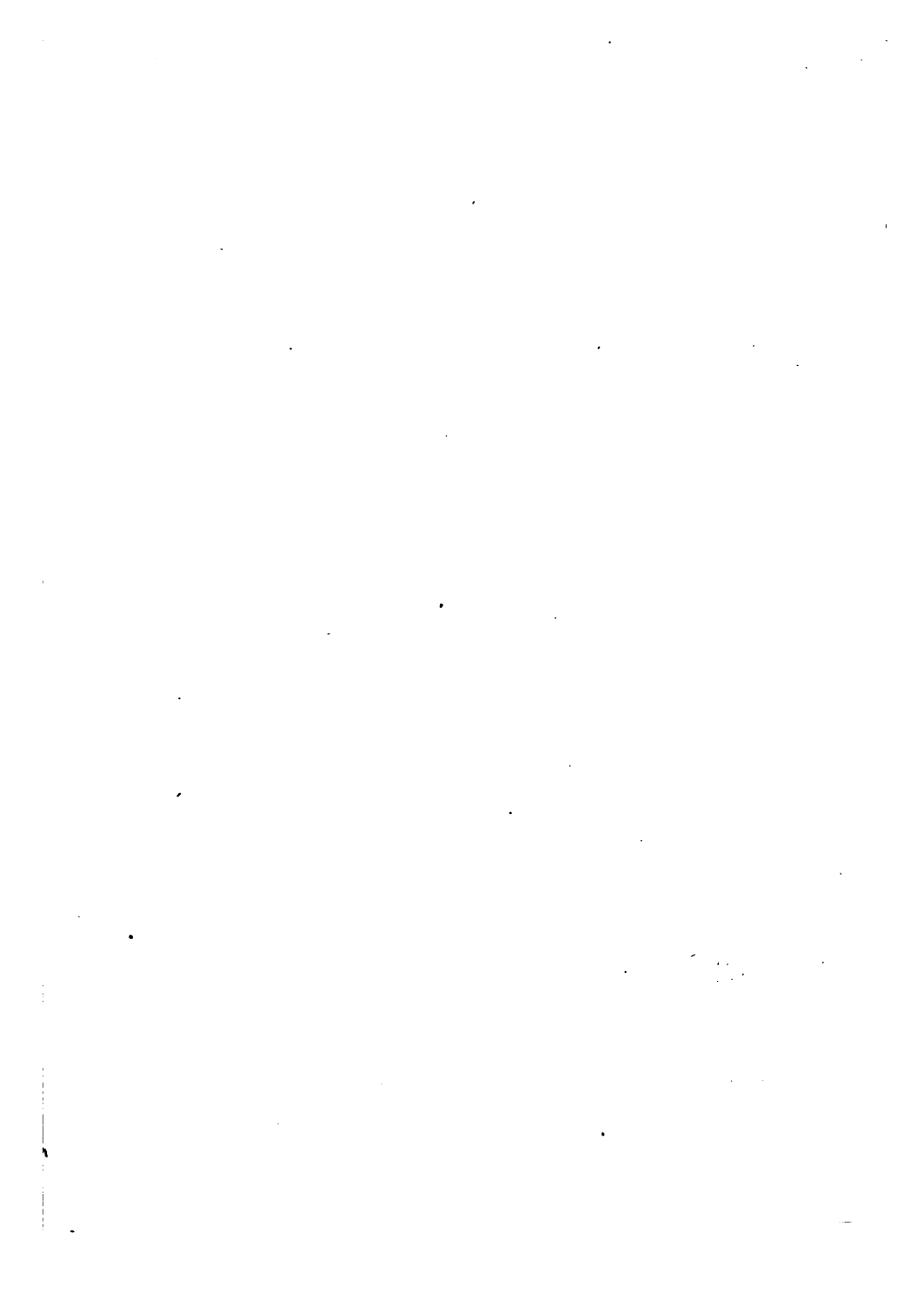
Prepare some dilute sulphuric acid by *slowly* pouring 15 cc. of concentrated acid into a beaker containing 50 cc. of water. Stir the water with a glass rod while the acid is being added. *Notice that the acid is poured into the water — never the reverse.* Cool the mixture and pour a few drops of it through the funnel tube. Hydrogen is at once evolved. Enough of the acid must be added from time to time to cause a gentle and continuous evolution of the gas. An excess of the acid should be avoided, however, or the action will become too violent, and a large quantity of zinc will have to be added at the close of the exercise. It is evident that the gas which passes over first is a mixture of hydrogen and air. *The student must remember that such a confined mixture of hydrogen and air explodes with great violence if ignited.* Hence see that the end of the delivery tube is not brought near any flame. Determine when the hydrogen is free from air by repeatedly collecting a test tube full of gas and igniting it, holding the tube mouth downward. If pure, the gas burns quietly; otherwise there is a slight explosion. After all the air has been expelled from the generator collect four bottles (250 cc., wide mouthed) of the gas. What is the source of the hydrogen? What is the use of the zinc? Why is the copper sulphate solution added? Remove the cork from the generator, add a few more pieces of zinc, and set aside until the next laboratory period. Sufficient zinc should be used so that at least a small portion of it remains undissolved.

II. PROPERTIES OF HYDROGEN

1. Thrust a lighted splint into a bottle of the gas held mouth downward. Slowly withdraw the splint and again thrust it into the gas. Describe the results. What do they prove?

2. Fill a small (60 cc.) wide-mouthed bottle or test tube one third full of water, and invert it in a pneumatic trough. Displace the remaining water with hydrogen. What does the bottle now contain? Withdraw it from the water and, holding it at arm's length, quickly bring it, mouth downward, over a flame. What do the results prove?

3. Uncover a bottle (mouth upward) of the gas. After a minute test for the presence of hydrogen with a lighted splint. Repeat, holding the bottle mouth downward. Describe the results. Is the gas heavier or lighter than air?



EXERCISE 6

I. COMPOUNDS FORMED IN THE PREPARATION OF HYDROGEN

Without removing the fragments of undissolved zinc, place the liquid obtained at the close of I, 2, Exercise 5, in an evaporating dish and boil gently on a ring-stand support. As soon as white crusts begin to form just above the liquid in the dish, filter the hot liquid into a beaker and set it aside to cool. How does the product which separates from the filtrate compare in properties with the original zinc? Has the zinc undergone a physical or a chemical change? What has become of it?

II. COMBUSTION OF HYDROGEN

Wash out the hydrogen generator used in I, 2, Exercise 5, and arrange it as shown in Fig. 16. The delivery tube *D* is

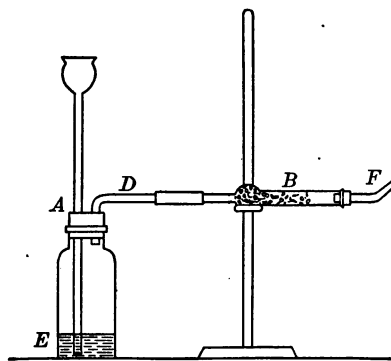


FIG. 16

connected with the drying tube *B* by a short piece of rubber tubing.

The drying tube is filled with calcium chloride, held in place by loose plugs of cotton placed at each end of the tube. *F* is a bent glass tube drawn out to a jet. After the apparatus has been approved by the instructor charge the generator with 6 or 8 g. of zinc, add the

solution of copper sulphate, cover with water, and add dilute sulphuric acid as in I, 2, Exercise 5. Slip a piece of rubber tubing over the tube *F* and test samples of the gas collected over water. *After all the air has been expelled, wrap a towel*

carefully about the generator and cautiously ignite the hydrogen. The flame is nearly colorless and is very hot. Test the heat of the flame by holding in it different objects, as a splint, a piece of picture-frame wire, a bit of charcoal.

III. THE OXYHYDROGEN BLOWPIPE

Examine the structure of the oxyhydrogen blowpipe. Draw a diagram representing a cross section of it. Compare it with the ordinary laboratory blowpipe (blast lamp). Why not have a short inner tube?

EXERCISE 7

QUALITATIVE SYNTHESIS OF WATER

1. Generate hydrogen as in II, Exercise 6. (*Note the precautions.*) Ignite the hydrogen and hold over the flame a cold, dry beaker. What substance condenses on the sides of the beaker? Account for its formation. The method of formation proves the presence of what elements in it?

2. Arrange an apparatus according to Fig. 17. *B* is a tube

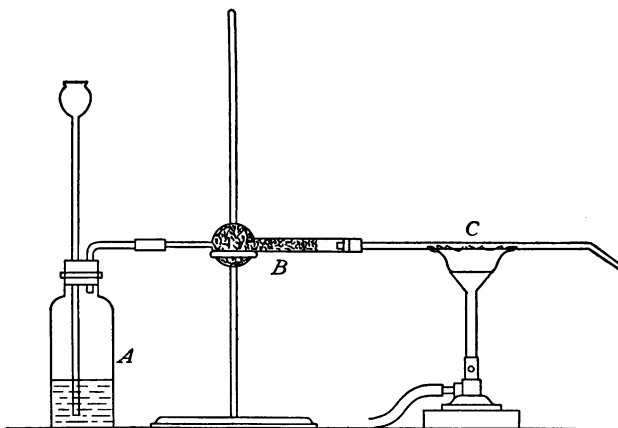


FIG. 17

filled with calcium chloride and *C* a hard-glass tube about 30 cm. long and from 8 to 10 mm. in diameter. Spread 1 g. of black copper oxide in a layer in *C*, 10 or 12 cm. from the end which is connected with *B*. This is done as shown in Fig. 13, except that the tube must be held in a horizontal position and the oxide deposited at the proper place by turning the paper over. Now generate hydrogen as in I, 2, Exercise 5. After all the air has been expelled from the apparatus and the generator wrapped in a towel, cautiously heat the copper oxide to redness with a "wing-top" burner. Note the condensation of moisture in the cold

portions of the tube. Account for its formation. What change has the copper oxide undergone? Is there any visible evidence of this change? Suggest a method of finding the weight of the water formed.

3. Without removing the contents of the hard-glass tube, replace the hydrogen generator and drying tube by the oxygen generator described in I, Exercise 4. Pass a *slow* current of oxygen through the tube, gradually heating its contents to redness. What change takes place? How does the product compare with the original copper oxide?



DISTILLATION

1. Connect a Liebig condenser with a 500-cc. flask, as represented in Fig. 18. The tube *C* is connected with the water pipe by means of rubber tubing, and a current of cold water is forced

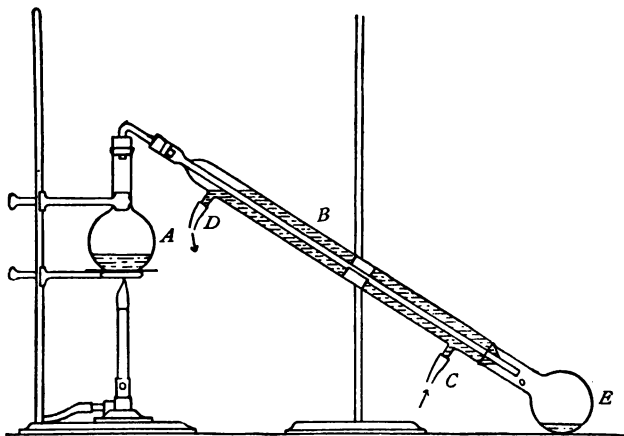


FIG. 18

through the outer tube of the condenser. (Why is cold water forced in at *C* rather than at *D*?) Half fill the flask with hydrant water and boil until 100 cc. or more liquid has collected in the receiver *E*. Is the change physical or chemical? Compare the *distillate* (distilled water) with the hydrant water in appearance and taste. To what is the difference due? Place four or five drops of the distilled water on a watch glass and evaporate, holding the watch glass 10 or 15 cm. above the tip of the flame. Is there any residue? Repeat, using hydrant water. Why is distilled water used in the laboratory?

2. Dissolve one or two small crystals of copper sulphate in 100 cc. of water and distill a few cubic centimeters. What is the composition of the distillate?

3. Distill a mixture of 10 cc. of alcohol (boiling point 78.3°) and 30 cc. of water. Collect the first 1 or 2 cc. of the distillate in an evaporating dish and test with a flame. In the same way test successive portions of the distillate. Does there seem to be a partial separation of the two liquids? In this way a mixture of liquids boiling at different temperatures may often be separated more or less perfectly. What name is given to this process?

I. WATER OF CRYSTALLIZATION

1. Heat some small crystals of zinc sulphate in a dry test tube. What evidence have you of the presence of water in the crystals? Examine the residue. How does it differ from the original crystals in form and composition?

2. Select some small crystals of copper sulphate. Do they appear to be dry? Fill a test tube one fourth full of these crystals, and heat as in 1. Compare the residue in form, color, and composition with the original crystals. Dissolve the residue in as little hot water as possible, pour the solution (note its color) into an evaporating dish, and set aside until crystals are deposited. Do these appear to be identical with the original crystals of copper sulphate?

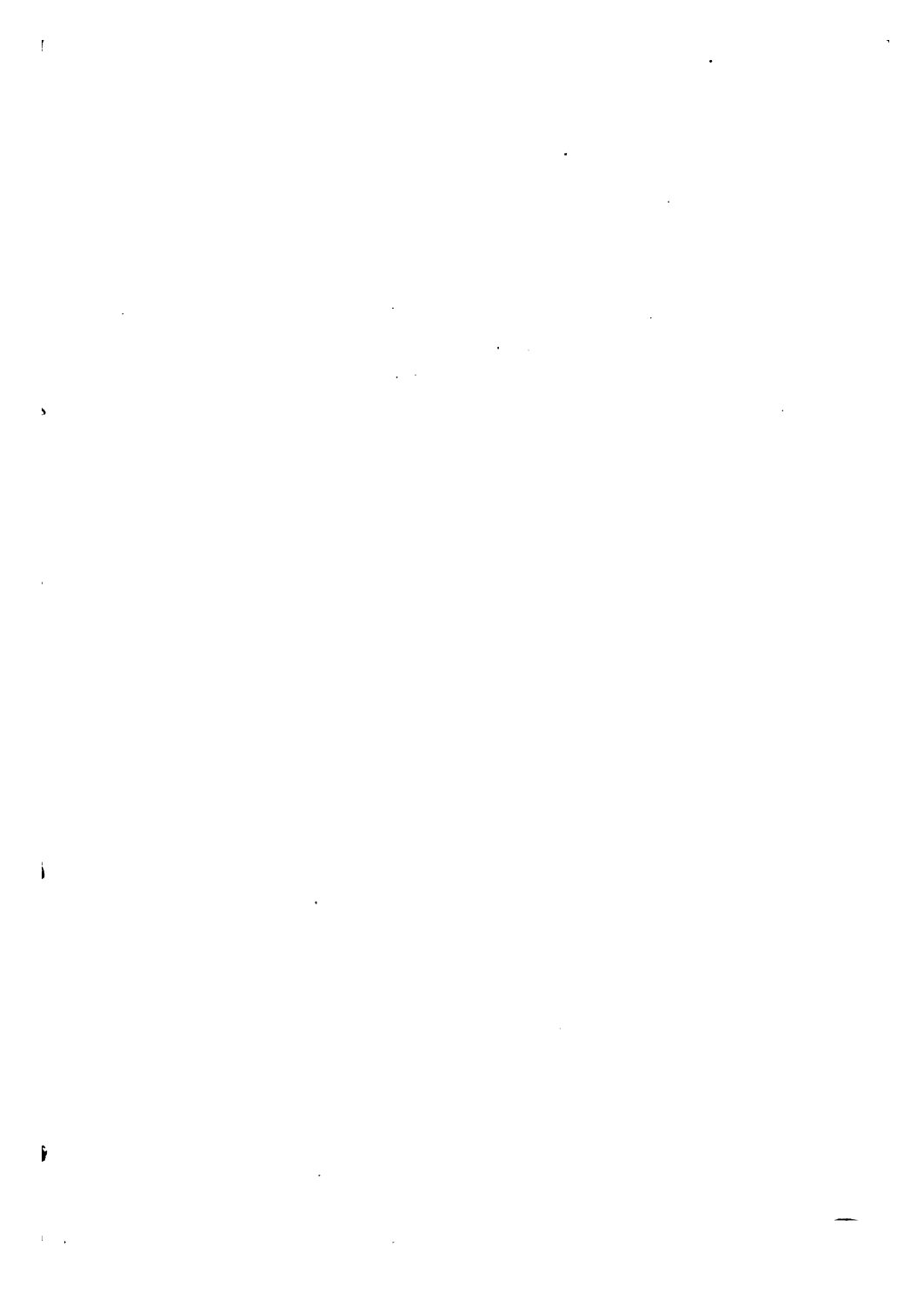
Does water seem to be necessary to the existence of the zinc and copper sulphates in the crystalline form? What is such water called? Is it combined with the substance composing the crystal or simply mixed with it? Give reasons for your answer. Do all crystals contain it? (Try a crystal of potassium dichromate.)

3. Expose a clear crystal of sodium sulphate to the air for one or two hours. Note the change in its appearance. To what is the change due? What are such bodies called?

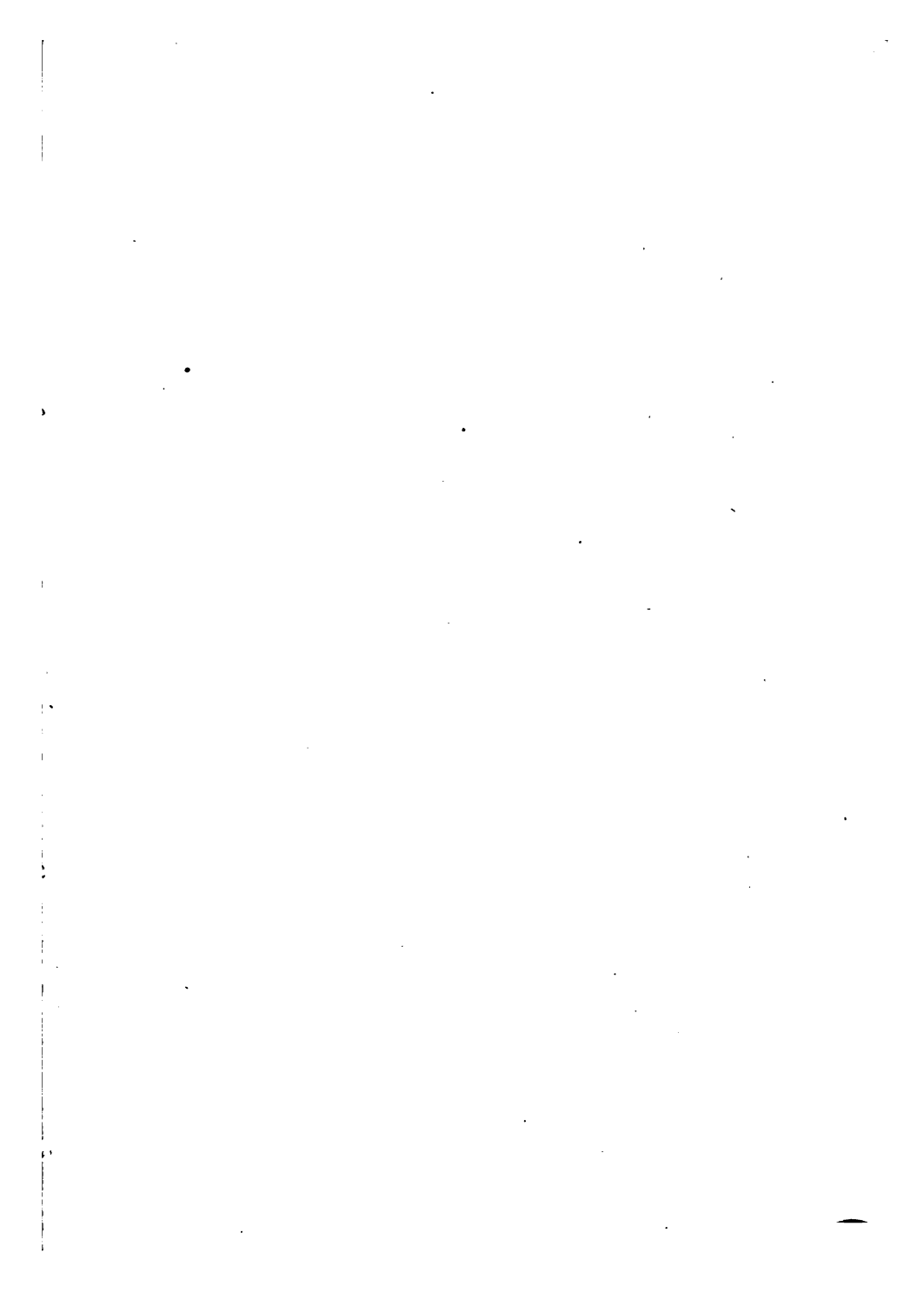
4. Place a small piece of calcium chloride on a watch glass and expose it to the air for two or more hours. Note the change. What term is applied to bodies that undergo this change?

II. DETERMINATION OF THE AMOUNT OF WATER OF CRYSTALLIZATION IN COPPER SULPHATE

Accurately weigh (or counterpoise) a porcelain crucible and cover. Then add 2 or 3 g. of *small* crystals of copper sulphate and again accurately weigh. Place the covered crucible on a



pipestem triangle and heat with a gentle flame until the color of the crystals has entirely disappeared. This will require from 20 to 30 minutes. The tip of the flame must not quite touch the crucible. The product is anhydrous copper sulphate. When the crucible is cool, reweigh. From your results calculate the percentage of water of crystallization in the crystals. Repeat the experiment, using a different weight of the crystals. Compare your results.



DETERMINATION OF THE AMOUNT OF COMMON SALT
OBTAINED BY ADDING HYDROCHLORIC ACID TO A
DEFINITE AMOUNT OF SODIUM BICARBONATE

When hydrochloric acid is added to sodium bicarbonate there are formed common salt (sodium chloride), water, and carbon dioxide. To determine the weight of salt formed from a definite weight of sodium bicarbonate proceed as follows: Carefully weigh an evaporating dish and watch glass. Transfer to the dish about 1 g. of sodium bicarbonate and weigh again. Pour 4 or 5 cc. of water on the bicarbonate, and place the watch glass on the dish so that only the lip of the dish remains uncovered. Now pour down the lip of the dish two or three drops of hydrochloric acid. Wait until the effervescence caused by the escape of the carbon dioxide ceases, and then add a few drops more of the acid. Repeat until the addition of the acid no longer causes any effervescence. Now hold the watch glass in the hand just above the dish and with a little water carefully rinse the liquid which has collected on the under surface of it back into the dish. Lay the watch glass aside and slowly evaporate the solution. The heat must be regulated so that the solution will not quite boil. Why?

When the solution has evaporated nearly to dryness, only a few drops of the liquid being left, cover the dish with the watch glass and increase the heat. The tip of the flame should just touch the bottom of the dish. Continue the heating until there is no more liquid left in the dish or clinging to the under surface of the glass. Then withdraw the heat and, after the dish is cool, reweigh. The residue is common salt. From your results calculate the amount of salt formed from 1 g. of the bicarbonate. Repeat the experiment, using a different weight of sodium bicarbonate. Compare the results of the experiments. Are they in accord with the law of definite composition?

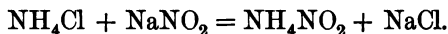


PREPARATION AND PROPERTIES OF NITROGEN

PRECAUTION. — Phosphorus must be kept and handled under water. Never touch the substance, as it may ignite and cause a dangerous burn.

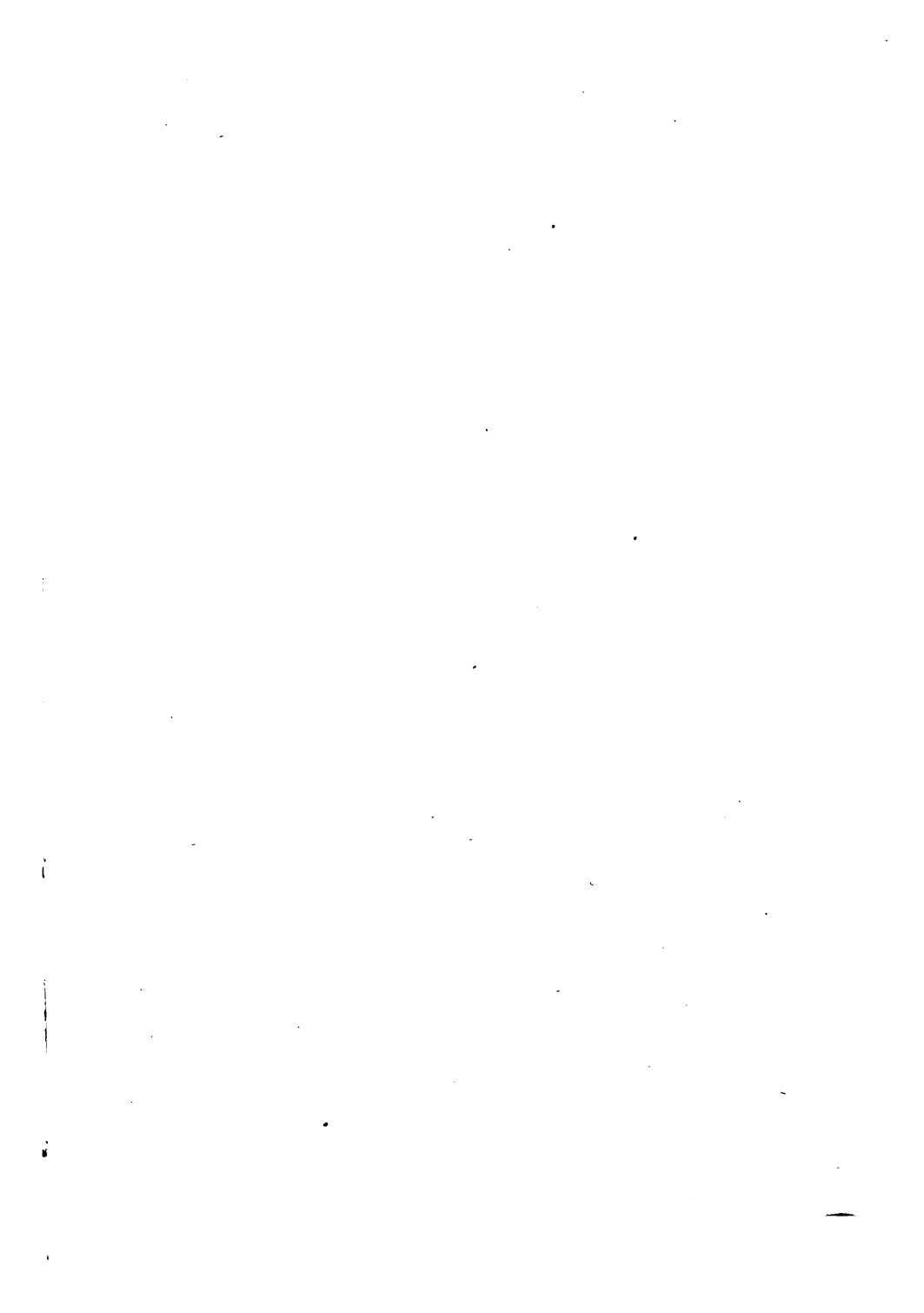
1. Cover the bottom of a pneumatic trough with water to a depth of 2 or 3 cm. On this float a porcelain crucible containing a small piece of phosphorus. Ignite the phosphorus by touching it with a hot wire, and quickly invert over the crucible a large beaker, being careful to keep the rim of the beaker below the surface of the water. Leave the beaker in position until the fumes have entirely disappeared. Note that the water has risen in the beaker. Explain. Adjust the beaker so that the level of the water inside and outside is the same; then cover the beaker with a glass plate and place it in an upright position. Test the gas with a burning splint.

2. Place in a 250-cc. flask a mixture of 3 g. of ammonium chloride and 6 g. of sodium nitrite, and add 20 cc. of water. Provide the flask with a cork (one-holed) and delivery tube so that the evolved gas may be collected over water, as in the case of oxygen and hydrogen. Have at hand a vessel of cold water so that the flask may be cooled by lowering it into the water in case the action becomes too violent. Clamp the flask and apply a *very gentle* heat, moving the burner about with the hand. As soon as the action begins withdraw the heat. After the air has been expelled from the apparatus fill two or three bottles (250 cc.) with the gas. If the action becomes too violent, immerse the flask in cold water. The reaction which takes place is expressed in the following equation :



The ammonium nitrite then decomposes into water and nitrogen (R).¹ Note the physical properties of the gas. Will it burn?

¹ The character (R) means that the equation is to be written by the student.



I. DETERMINATION OF THE RELATIVE AMOUNTS OF NITROGEN AND OXYGEN IN THE AIR (FROM COOLEY'S LABORATORY STUDIES)

This may be done by bringing in contact with a definite volume of air a liquid which not only absorbs the oxygen, but in doing so flows into the tube which contains the air and fills a space equal to that previously occupied by the oxygen. The volume of this liquid can be easily measured, and hence the volume of the absorbed oxygen ascertained.

The solution used to absorb the oxygen soon loses its strength on exposure to air; hence the experiment must be performed rapidly. Before preparing the solution the student should practice the manipulations involved in the experiment.

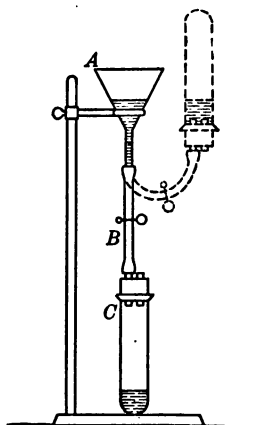
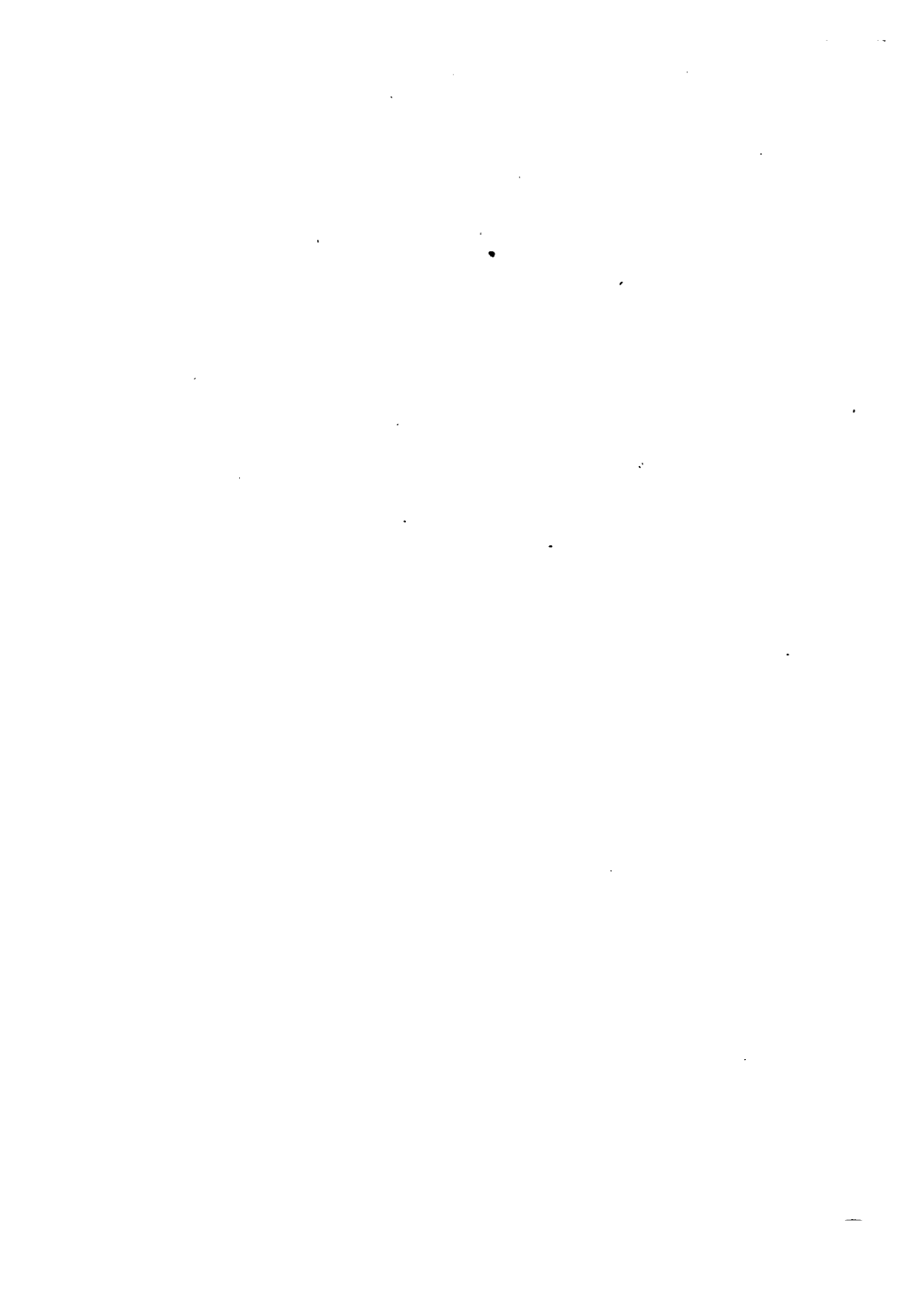


FIG. 19

Prepare an apparatus according to Fig. 19. *C* represents a test tube about 15 cm. in length (use the hard-glass tube employed in the preparation of oxygen). The tube is fitted with a two-holed rubber stopper. One hole is closed with a glass rod while the other is fitted with a small glass tube, the upper end of which extends 4 or 5 cm. above the stopper. A piece of soft rubber tubing 12 or 15 cm. in length connects the glass tube with a small funnel, as shown in the figure. Now close the rubber tube tightly with the screw clamp. Disconnect the test tube and remove

the glass rod from the stopper preparatory to performing the experiment. Prepare an alkaline solution of pyrogalllic acid, as follows: Dissolve 5 g. of potassium hydroxide in 5 cc. of water, and cool the solution to the temperature of the room. Add



this to a solution of 4 g. of pyrogalllic acid in 10 cc. of water, and *at once* pour the resulting liquid into the funnel *A*. Quickly open the screw clamp until both the rubber and glass tubes are filled with the liquid, and then close tightly. Connect the test tube, holding it by the rim to avoid heating the contained air, and insert the glass rod in the cork. The air inclosed in the tube is now at the same temperature and pressure as the surrounding air. Now open the screw clamp. The liquid flows in, absorbing the oxygen. When the liquid ceases to enter, grasp the tube by the rim and invert it, as shown by the dotted lines of the figure, adjusting it so that the level of the liquid is the same in both the tube and funnel. Why? Then clamp the rubber tube and return the test tube to its original position. Mark the volume of the air originally inclosed in the tube by placing a narrow strip of gummed paper about the tube at the lower end of the stopper; also the volume of the oxygen absorbed, by a strip of paper placed at the level of the liquid in the tube. Disconnect the tube and rinse it. Measure the volume of the tube to each strip of paper by pouring in water from a graduated cylinder. From these measurements calculate the number of volumes of oxygen and nitrogen in 100 volumes of air.

NOTE.—This experiment disregards the presence in the air of all constituents other than oxygen and nitrogen. The volume of such constituents, however, in the amount of air taken is smaller than the unavoidable errors in the experiment.

II. OTHER CONSTITUENTS OF THE AIR

1. Expose a piece of calcium chloride to the air for two or more hours. What do the results prove?
2. Expose a few cubic centimeters of limewater to the air for a half hour (R).

SOLUTIONS

1. Drop a crystal of potassium permanganate into a test tube partially filled with water and shake the liquid until solution takes place. Note the color of the solution.

2. Fill a test tube with water and set it in the test-tube rack. Drop into it a crystal of potassium permanganate and allow the liquid to remain until near the close of the laboratory period. Note the result. What does the experiment show?

3. Prepare a saturated solution of common salt in boiling water. Cool the solution to room temperature and filter off any undissolved solid. Weigh a small evaporating dish and watch-glass cover. Pour 10 or 15 cc. of the clear salt solution into the dish and reweigh. Now evaporate to dryness, taking note of the precautions given in Exercise 10. When the dish is cool reweigh. From your results calculate the solubility of common salt.

4. In a similar way determine the solubility of calcium sulphate and potassium dichromate.

5. Pour about 5 cc. of water into a test tube, add an equal volume of kerosene, and mix the liquids by shaking the tube. Set the tube aside for a few minutes and note the result. Repeat the experiment, substituting alcohol for the kerosene. Note the results.

I. ACIDS

Prepare a dilute solution of each of the following acids by adding one or two drops of the acid to 10 cc. of water: hydrochloric, nitric, sulphuric, acetic. By means of a clean glass rod transfer a drop of each to a piece of blue litmus paper. Note the result. In a similar way try their effect on red litmus paper. Taste a drop of the dilute solutions. Compare the formulas of the acids. In what respect are they similar in composition?

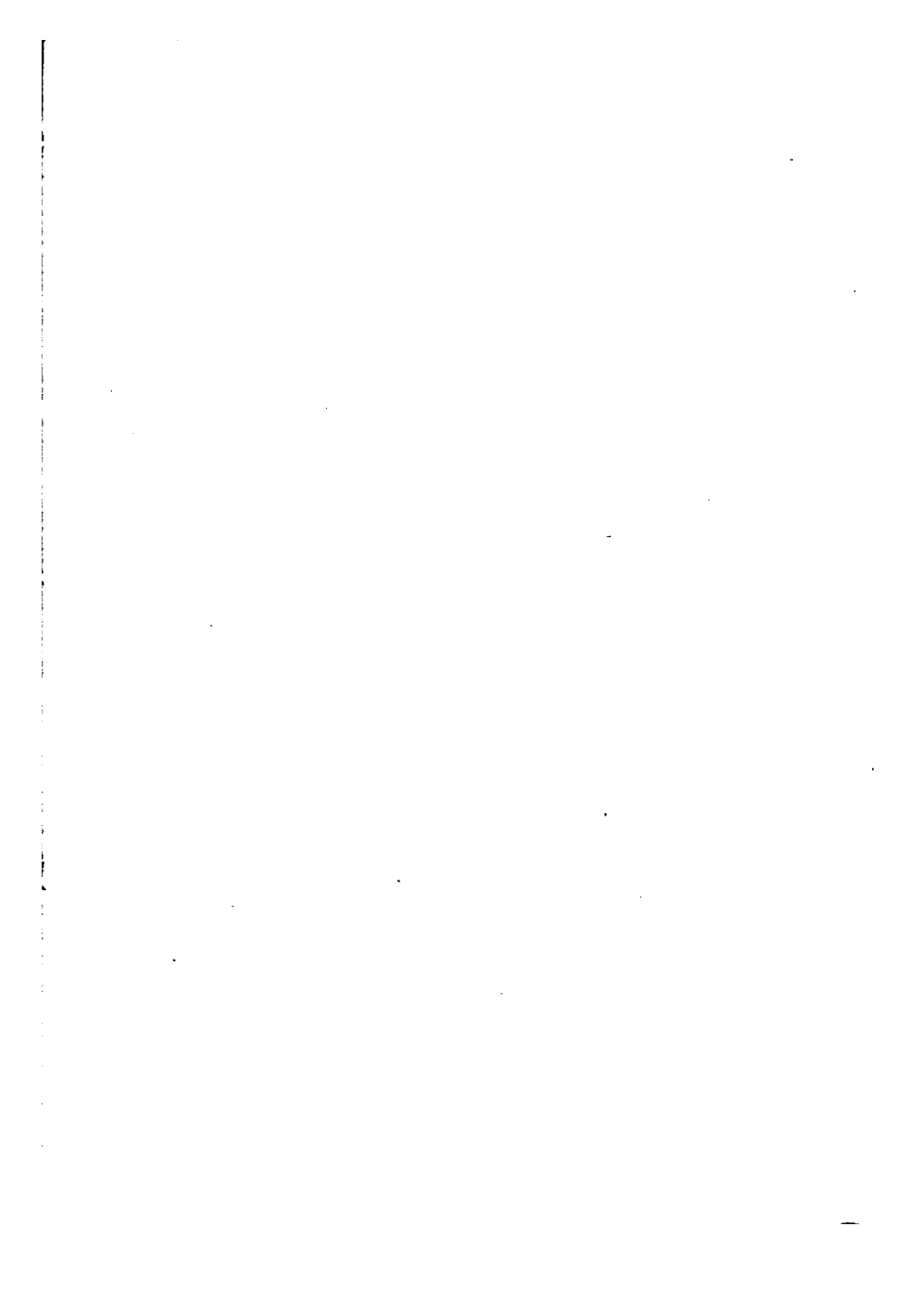
II. BASES

In a similar way try the effect on red litmus paper of a solution of each of the following bases: sodium hydroxide, potassium hydroxide, calcium hydroxide. Do they affect the blue litmus paper? Taste a drop of the calcium hydroxide solution. Compare the formulas of the bases. In what respect are they similar in composition?

III. INTERACTION OF ACIDS AND BASES — SALTS

1. Dilute 5 cc. of the ordinary laboratory solution of sodium hydroxide (1 part of the base to 10 parts of water) with an equal volume of water. To this solution add a few drops of hydrochloric acid (R). Stir the resulting solution with a glass rod and test its action on blue and red litmus paper. Has it acid or basic properties? Now continue to add the acid drop by drop until the resulting solution is neutral (i.e. has no effect on either blue or red litmus paper) or is at most slightly acid. Pour the solution into an evaporating dish and evaporate to dryness. What compound remains? Taste it. What is the name given to the compounds formed by the interaction of acids and bases?

2. Characterize acids and bases, first, as to composition; second, as to their interaction with each other; third, as to their action on litmus; fourth, as to taste.



RATIO OF ACID TO BASE IN NEUTRALIZATION

Prepare a dilute solution of sodium hydroxide by diluting 20 cc. of the laboratory reagent to 100 cc. ; also a dilute solution of sulphuric acid by adding 1 cc. of the strong acid to 100 cc.

of water. Rinse out a burette, first with distilled water and then with a little of the alkaline solution. Support the burette (Fig. 20), and pour into it the alkaline solution until the level of the liquid is 2 or 3 cm. above the zero mark. Turn the stopcock and let the solution flow out until the bottom of the curved surface (meniscus) is on a level with the zero mark.

In a similar way fill a second burette with the acid solution. Now let exactly 15 cc. of the acid solution flow into a small beaker, add two drops of phenolphthalein solution, and run in 2 or 3 cc. of the alkaline solution. Notice that where the liquids come in contact a reddish color

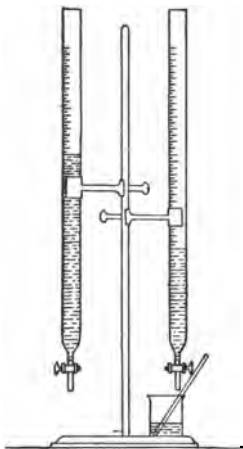
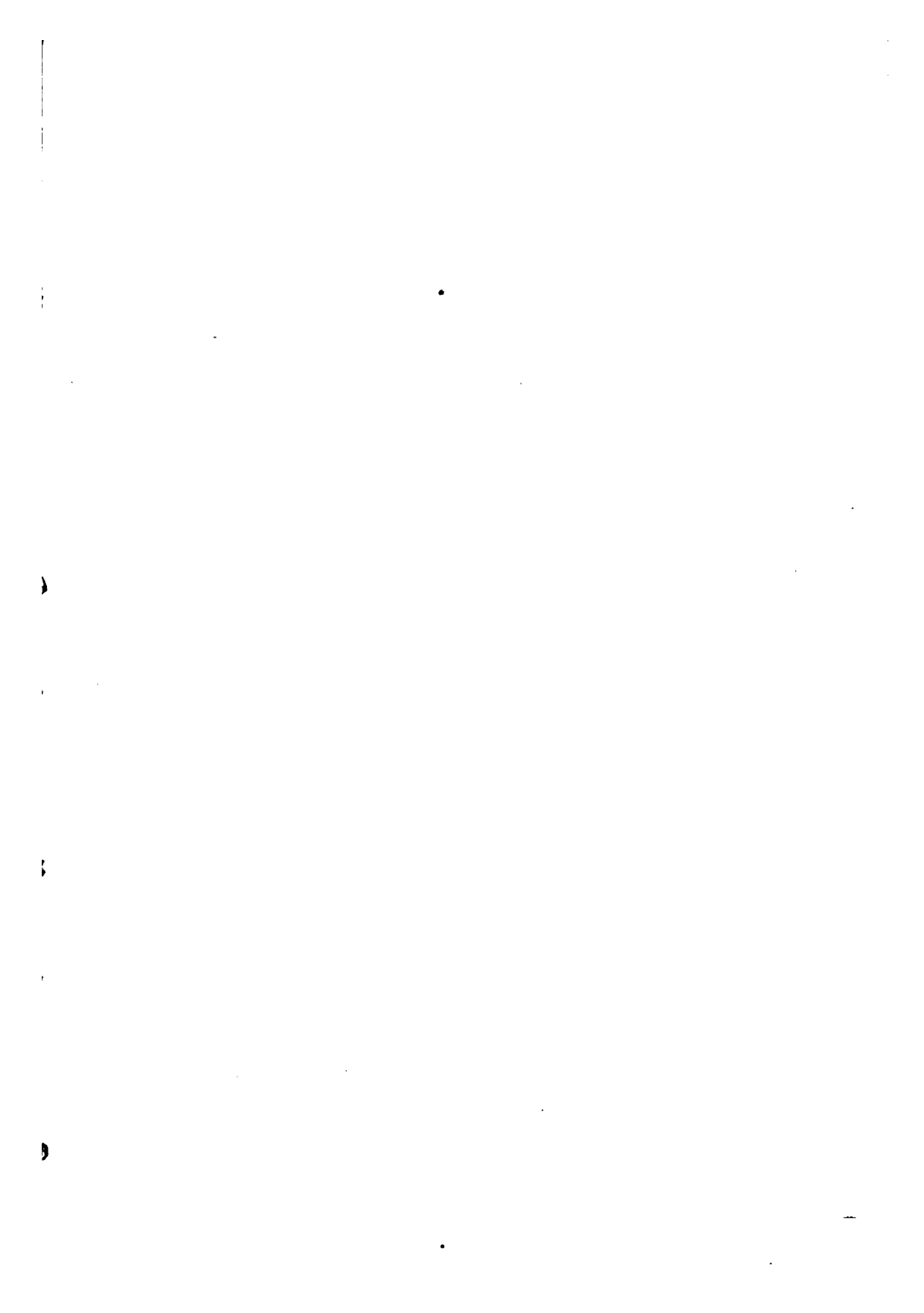


FIG. 20

is produced, which disappears quickly on stirring. Run in more of the solution, a little at a time, until the color fades slowly, and then a drop at a time until the entire liquid, on stirring, remains colored *faintly* pink. This marks approximately the point of neutralization. Note the number of cubic centimeters of the alkaline solution used. Repeat the experiment, using different amounts of acid, say 10 cc. and 20 cc. Calculate in each case the number of cubic centimeters of the alkaline solution required to neutralize 1 cc. of the acid solution. What do the results prove?



I. PREPARATION OF AMMONIA

1. Dissolve 1 g. of ammonium chloride in a little water in a test tube and heat to boiling. Can you detect the odor of ammonia? Add a few drops of a solution of sodium hydroxide. Is the ammonia liberated (R)? Moisten a small strip of red litmus paper and hold it at the mouth of the tube. Note the result. Hold the end of a glass rod moistened with strong hydrochloric acid in the mouth of the tube. What is formed (R)?

2. *Usual laboratory method.* This differs from the method used in 1 only in the fact that the less expensive calcium hydroxide (slaked lime) is substituted for the sodium hydroxide.

The form of apparatus used is shown in Fig. 21. *A* represents a 250-cc. flask. *B* and *C* are 250-cc. wide-mouthed bottles partially filled with water. Notice that the glass tubes extending into the bottles do not quite touch the surface of the water. Two *dry* bottles will also be needed for collecting some of the gas.

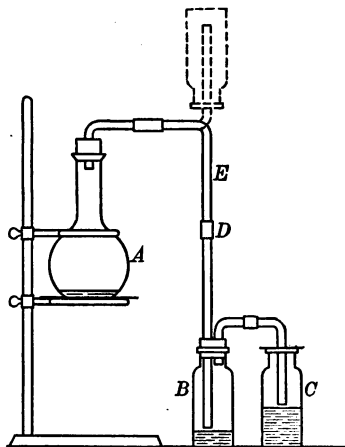


FIG. 21

Put into the flask *A* an intimate mixture of about 30 g. of finely powdered slaked lime, 15 g. of ammonium chloride, and sufficient water to form a thick paste; place the flask on a

sand bath and heat gently with a *small* flame. A wire gauze may be substituted for the sand bath, provided the flask is clamped so that the bottom does not quite touch the gauze. In this case, however, the burner should be held in the hand and moved about so as to apply the heat uniformly; otherwise

EXERCISE 16 (continued)

the flask may be broken. As soon as the gas is evolved freely, disconnect the tube *E* at *D*, bring it to an upright position, as shown in the dotted lines, and collect two bottles of the gas by bringing them successively down over the exit tube. To tell when they are filled, test for the presence of ammonia at the mouths of the bottles with a piece of red litmus paper moistened with water. As soon as filled, cover the bottles tightly and set them aside, mouth downward; then quickly connect the tube *E* with the bottle *B* again and continue to heat the mixture gently as long as any gas is generated. What is the source of each of the materials used in the preparation of the gas? Write the equations for all the reactions involved.

II. PROPERTIES OF AMMONIA

1. Note the color and odor of the gas. Is it heavier or lighter than air?
2. Test a bottle of the gas with a burning splint. Describe the results.
3. Fill a large beaker with water and color it with a few drops of red litmus solution. Uncover the remaining bottle of the gas and at once bring its mouth under the surface of the water in the beaker. What do the results prove?
4. Note the odor of the liquid in the bottle *B*. Try its effect on blue and red litmus papers. How does it compare with the "aqua ammonia" of the druggist? Does the gas combine with the water or is it simply dissolved in it? Give reasons for your answer. Now neutralize the liquid with hydrochloric acid (R) and evaporate just to dryness. Test the residue with a few drops of sodium hydroxide solution (R).



I. PREPARATION OF NITRIC ACID

Arrange an apparatus as shown in Fig. 22. The retort should have a capacity of from 100 to 150 cc. Put in the retort about 12 g. of sodium nitrate and 10 cc. of sulphuric acid, pouring the latter through a funnel placed in the tubulure of the retort. Heat the mixture gently with a small flame. Nitric acid is generated (R), distills over, and is condensed in the test tube, which must be kept cool with water.

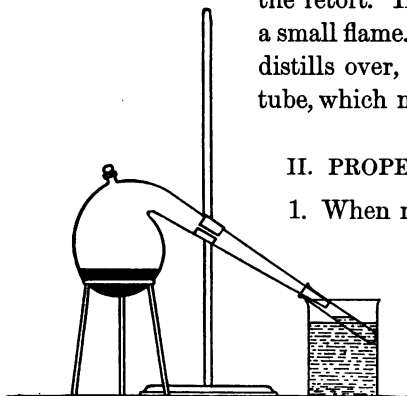


FIG. 22

II. PROPERTIES OF NITRIC ACID

1. When nitric acid is heated a part of it is decomposed into water, nitrogen peroxide, and oxygen (R); hence it is a good oxidizing agent. To test this, put a small piece of tin in a test tube, cover it with a little nitric acid, and

heat gently (hood). The white residue formed is composed mainly of tin and oxygen, the latter being taken from the nitric acid.

2. Pure nitric acid is colorless. How do you account for the color of the acid which you have prepared?

3. Dissolve a crystal of sodium nitrate in 2 or 3 cc. of water in a test tube, add an equal volume of sulphuric acid, and cool. Now gently pour a concentrated solution of ferrous sulphate down the side of the tube so that it floats on the heavier liquid. A brown ring soon forms where the liquids meet. Repeat the experiment, using potassium nitrate. This is a good test for nitrates. The brown ring is due to the formation of a compound of ferrous sulphate and nitric oxide, the latter being formed by the reduction of the nitric acid by the ferrous sulphate.



PREPARATION AND PROPERTIES OF SOME OF THE
OXIDES OF NITROGEN

1. *Nitrous oxide.* Put 6 or 8 g. of ammonium nitrate in the hard-glass test tube used in the preparation of oxygen. Attach a delivery tube and heat *gently*, applying no more heat than is necessary to cause a gentle evolution of the gas. As soon as the gas is regularly evolved, collect two or three bottles of it over water. Notice the water deposited on the sides of the test tube. What is the source of it (R)? Note the color, odor, and taste of the gas. Test it with a glowing splint. Account for the result. How can you distinguish nitrous oxide from oxygen?

2. *Nitric oxide and nitrogen peroxide.* Put a few pieces of copper in your hydrogen generator (hood), just cover them with water, and add 2 or 3 cc. of nitric acid. Collect over water two bottles of the evolved gas, adding more nitric acid if necessary. Compare the color of the gas in the generator with that collected in the bottles and account for any difference. Write the equations for all the reactions involved. Uncover one of the bottles containing gas and account for the result (R). Test the gas in the second bottle with a burning splint. Which is the more stable, nitrous oxide or nitric oxide? Give reasons for your answer.



PROPERTIES OF SULPHUR

1. Examine the physical properties of a piece of brimstone. Pour 2 or 3 cc. of carbon disulphide (*keep away from flame*) over a little powdered brimstone in a test tube. Cover the mouth of the tube with the thumb and shake the contents gently until the sulphur is dissolved. Pour the clear solution into an evaporating dish, cover it loosely with a filter paper, and set it aside in the hood. The carbon disulphide soon evaporates, the sulphur being deposited in crystals. Examine these with a magnifying glass.

2. Half fill a test tube with powdered brimstone and apply heat enough to just melt it. Note the properties of the liquid. Now apply a stronger heat and observe that the liquid becomes darker and at a certain temperature (200° – 250°) is so thick that the tube may be inverted without spilling it. Finally increase the heat until the sulphur boils (448°) and pour the boiling liquid into a beaker of cold water. Examine the product. What name is given to this form of sulphur? Expose it to the air for an hour. Have its properties remained unchanged?

3. Fill a porcelain crucible with powdered brimstone and apply a very gentle heat until the sulphur is just melted. Withdraw the flame and examine the liquid carefully as it cools. Crystals soon begin to form on the surface, rapidly extending in from the circumference toward the center. Before they reach the center, quickly pour off the remaining liquid and examine the crystals. Contrast these with those formed in 1. In how many forms have you obtained sulphur?

4. Burn a small piece of sulphur. What is the product of combustion? Note its odor. Boil a little sulphur in the test tube used in 2, and drop a small strip of hot copper foil into the boiling liquid. Is there any visible evidence of a chemical change? What is formed? What is the product formed in II, 2, Exercise 2?

EXERCISE 20

PREPARATION AND PROPERTIES OF HYDRO-
SULPHURIC ACID

1. (Hood.) Attach a delivery tube to the hydrogen generator, as represented in Fig. 23. Put into the generator a few pieces of iron sulphide (FeS) and insert the stopper. Before generat-

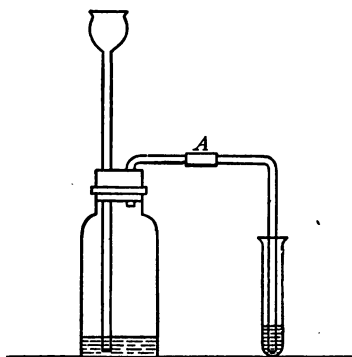


FIG. 23

ing the gas have at hand a dry bottle in which to collect some of it; also test tubes each about one fifth full of a solution of one of the following compounds: copper sulphate, zinc sulphate, cadmium chloride, magnesium sulphate, sodium chloride. Now pour a few drops of a dilute solution of sulphuric acid (1 part acid to 7 parts of water by volume) through the funnel tube of the generator, adding more from

time to time, if necessary, to maintain a gentle evolution of the gas (R). Collect a bottle of the gas by displacement of air, allowing the gas to flow in until it is ignited by a flame held at the mouth of the bottle (R). Account for the deposit on the sides of the bottle. Cause the gas to bubble for a few seconds through each of the solutions in the test tubes. Note the color of the precipitates obtained in the test tubes. Write the equations for all the reactions involved. Which ones of the solutions fail to give a precipitate? How do you account for this?

2. Pass a little of the gas into some water in a small beaker. How does the solution compare with the so-called "sulphur water" of many springs? Note the odor of the water. Drop a silver coin into it and account for the results. Why do certain foods, as eggs, blacken silver spoons?



I. SULPHUR DIOXIDE AND SULPHUROUS ACID

(Hood.) Place about 10 g. of copper turnings or small pieces of sheet copper in a generator arranged as in Fig. 24. Add about 25 cc. of concentrated sulphuric acid and apply a gentle

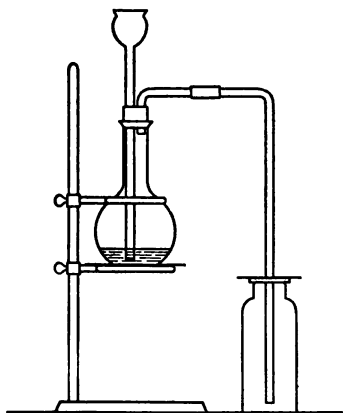
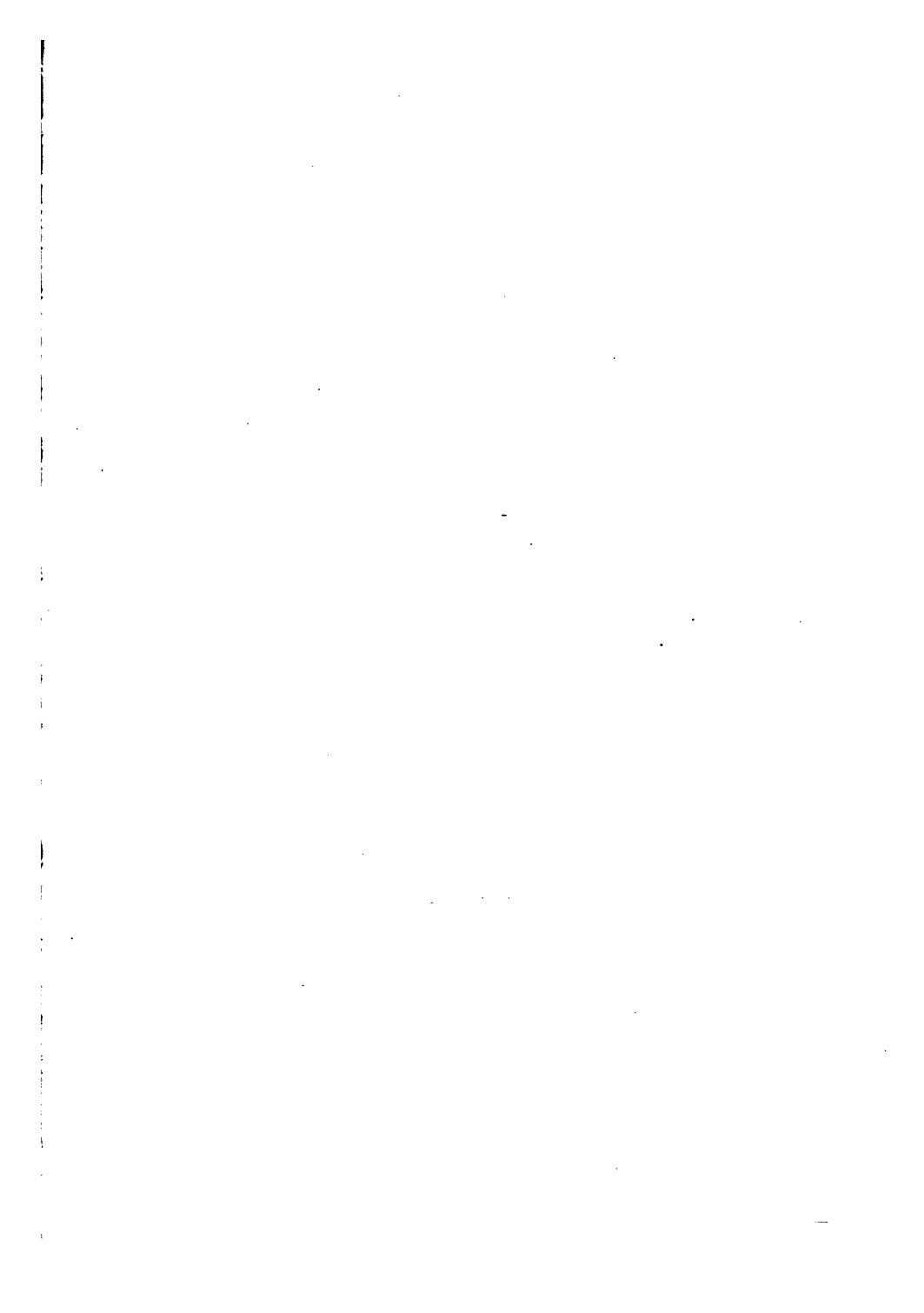


FIG. 24

heat. As soon as the action begins (R) lower the flame, regulating it so as to obtain a uniform evolution of the gas. Collect two bottles of the gas by displacement of air; then cause it to bubble through 25 cc. of water as long as any is dissolved. By what other method has this gas been prepared in a previous exercise? Invert one of the bottles of the gas so that its mouth is under water, and examine after several minutes.

Account for the results. Contrast the action of hot concentrated sulphuric acid on copper with that of the dilute acid on zinc and iron (R).

Test the saturated aqueous solution of sulphur dioxide with blue litmus paper. Is the gas combined with the water or simply dissolved in it? Give reason for your answer. Boil a few cubic centimeters of the solution in a test tube until the odor of the gas disappears, and then test with blue litmus. How do you account for the result? Set aside 5 cc. of the solution for a future experiment. To the remainder of the liquid add a solution of sodium hydroxide, drop by drop, until neutral (R), and evaporate just to dryness. What is the residue? Moisten it with two or three drops of hydrochloric acid. Note the odor of the evolved gas. What is it? How could you test for a sulphite?



II. SULPHURIC ACID AND SULPHATES

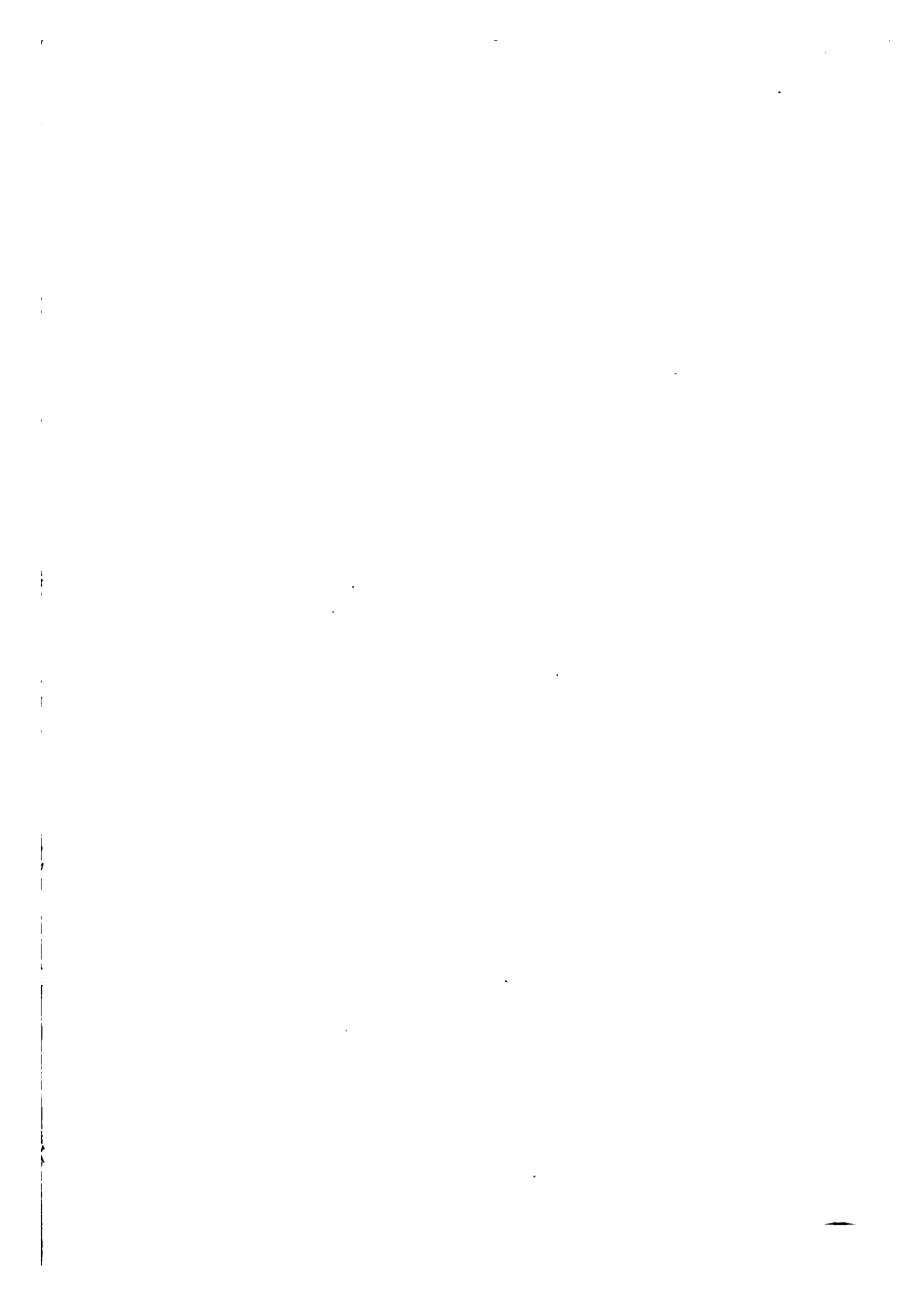
1. Heat a small bit of charcoal the size of a pea with 1 or 2 cc. of sulphuric acid. Note the odor of the evolved gas. What is it (R)? Under what conditions does sulphuric acid act as an oxidizing agent? Write in steps the equations for the reactions which take place when copper is acted on by nitric acid and hot concentrated sulphuric acid respectively, pointing out the similarity in the action of the two acids.

2. Put a drop of strong sulphuric acid on a splint. Pour a few drops on $\frac{1}{2}$ g. of sugar in a test tube. Examine after a few minutes. Account for the results.

3. Add three drops of sulphuric acid to 5 cc. of water in a test tube. To this add a few drops of a solution of barium chloride. Note that a precipitate forms (R). Now add three or four drops of hydrochloric acid. Does the precipitate dissolve? The formation of a precipitate with barium chloride, *which is insoluble in hydrochloric acid*, constitutes a good test for sulphuric acid.

4. Place 5 cc. of the solution of sulphur dioxide, obtained in I, in a test tube and add 1 cc. of concentrated nitric acid. Allow the solution to stand for five minutes and then test with barium chloride solution. Is sulphuric acid present (R)?

5. *Salts of sulphuric acid, — sulphates.* Dissolve in a little water a crystal of each of the following: sodium sulphate, magnesium sulphate, copper sulphate. Apply to each the barium chloride test for sulphuric acid (R). Can sulphates be detected by this method? Dissolve a crystal of sodium sulphite in water and apply the same test. How can you distinguish between sulphates and sulphites? (Sulphites very often contain more or less of the corresponding sulphates, due to the absorption of oxygen from the atmosphere.)



I. PREPARATION AND PROPERTIES OF HYDROFLUORIC ACID

PRECAUTION. — The acid is very corrosive and must not be inhaled; neither must its solution be brought in contact with the skin.

Place some pieces of paraffin on a glass plate and gently warm over a small flame. When the paraffin is melted tilt the plate about so as to form a uniform layer of the wax. When cold scratch your name *through* the wax with a pin or other sharp point. Place 3 g. of fluorspar in a lead dish and add sufficient sulphuric acid to make a paste of it. Cover the dish tightly with the waxed side of the glass plate, and set it in the hood for an hour; then scrape off the paraffin and examine the glass. Write the equations for all the reactions involved.

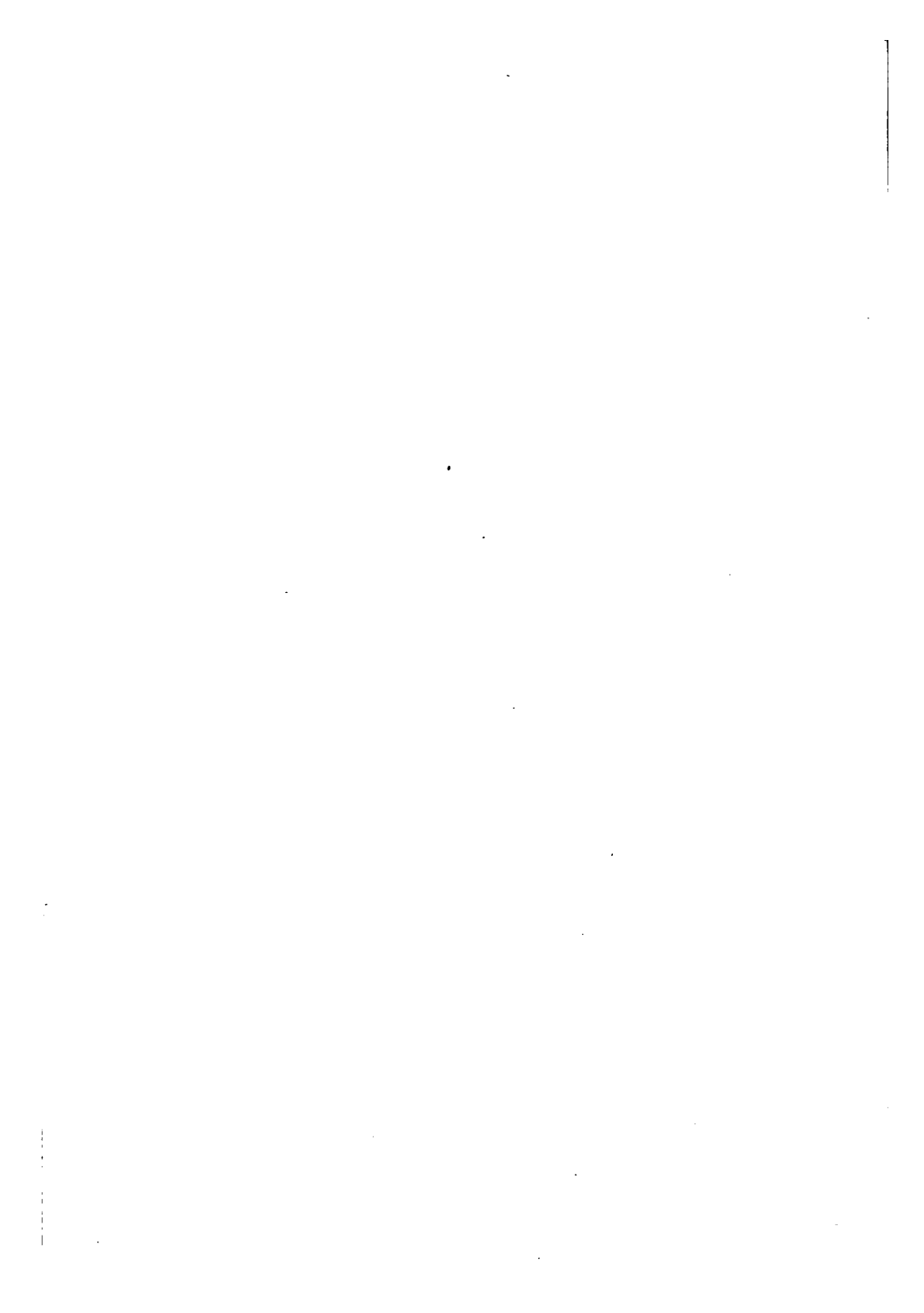
II. PREPARATION AND PROPERTIES OF CHLORINE

PRECAUTION. — All of the following experiments must be performed in the hood, and great care must be taken not to inhale the gas.

1. Place about 1 g. of manganese dioxide in a test tube, add 2 or 3 cc. of hydrochloric acid, and heat gently (R).

2. Repeat the experiment, replacing the hydrochloric acid with small amounts of common salt and sulphuric acid (R). The manganese dioxide and salt should first be mixed together and the sulphuric acid then added.

3. *Usual laboratory method* (two students working together). Arrange an apparatus according to Fig. 25, using a 250-cc. flask. Thoroughly clean and dry five wide-mouthed bottles (250 cc.) for collecting the gas. Place in the flask from 20 to 25 g. of manganese dioxide. Insert the cork and pour 150 cc. of hydrochloric acid through the funnel tube. Shake the flask so as to mix the contents thoroughly. Warm gently, applying just enough heat to cause a gentle evolution of the gas, but not



EXERCISE 22 (continued)

sufficient to boil the liquid. Fill all the bottles with the gas, collecting it by displacement of air; then prepare some chlorine water by bringing the exit tube into a bottle containing a little water so that the gas bubbles up through the liquid. Continue the gentle heating until no more chlorine is evolved.

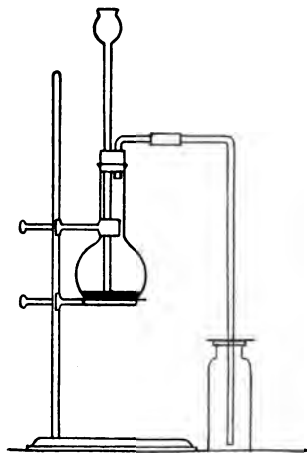


FIG. 25

water by bringing the exit tube into a bottle containing a little water so that the gas bubbles up through the liquid. Continue the gentle heating until no more chlorine is evolved.

4. Grind a fragment of antimony to a fine powder and sprinkle a pinch of the powder into one of the bottles of the gas. SbCl_3 is formed (R). Is the presence of oxygen necessary for combustion?

5. Support by forceps a small piece of copper foil, heat it to redness, and *immediately* thrust it into a bottle of the gas. Describe the result. What is formed?

6. Suspend a strip of colored calico in a bottle of the dry gas; also two strips of paper, the one with writing (ink) on it, the other with printing (printer's ink).

7. Repeat 6, using similar strips, but moistened with water. Describe the results in 6 and 7. What part does the water play in the bleaching?

PREPARATION AND PROPERTIES OF HYDROCHLORIC ACID

1. *Usual laboratory method.* Connect the generator used in the preparation of chlorine (Fig. 25) with a wide-mouthed bottle, as shown in Fig. 26. The delivery tube is cut at *A* and

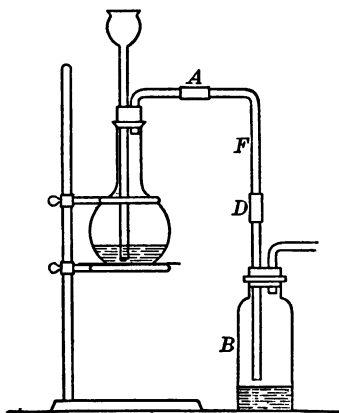


FIG. 26

D and the ends (rounded) joined by rubber tubing. Pour about 20 cc. of water into the bottle *B*. Notice that the tube extending into the bottle *does not quite touch* the surface of the water. Prepare some dilute sulphuric acid by carefully (refer to I, 2, Exercise 5) pouring 30 cc. of the concentrated acid into 10 cc. of water. The mixture must be stirred and cooled from time to time while the acid is being added. Have at hand two *dry* bottles in which to collect some

of the hydrochloric acid gas. Put about 50 g. of common salt into the generator flask, insert the cork, pour the cold, dilute sulphuric acid through the funnel tube, mix the contents by a gentle motion of the flask, and after two or three minutes warm gently with a *small* flame. Notice the currents in the water in *B*. What causes them? As soon as the gas is evolved regularly disconnect the generator flask at *D* long enough to collect two bottles of the gas by displacement of air. Cover these tightly with glass plates and set aside; then connect the generator with *B* again and continue to apply a gentle heat as long as any gas is evolved (R). Why not extend the tube in *B* to the bottom of the bottle?

2. What is the color of the gas? (Examine that in the generator.)

3. Test the gas in one of the bottles with a lighted splint. Is it combustible? Is it a supporter of combustion?

4. Fill a large beaker with water and color it with a few drops of a solution of blue litmus. Now uncover the remaining bottle, invert it, and at once bring its mouth under the surface of the water in the beaker. Describe the results. What does the experiment prove?

5. Put a drop of the aqueous solution of the acid from bottle *B* on a bit of blue litmus paper. Note the result. Pour two drops of the solution into 3 or 4 cc. of water and taste a drop. Perform a test-tube experiment to prove the presence of chlorine in the acid (R); also one to prove the presence of hydrogen (R). How does the solution compare with the hydrochloric acid on your desk?

I. BROMINE

PRECAUTION. — The vapor of bromine must not be inhaled. Perform the experiments in a hood.

The preparation and properties of bromine. Use the apparatus employed in the preparation of nitric acid (Fig. 22). Put into the retort a mixture of 2 g. of potassium or sodium bromide and 4 g. of manganese dioxide, and add to this through a funnel a cold dilute solution of sulphuric acid, prepared by slowly adding 10 cc. of sulphuric acid to 40 cc. of water. Shake the retort so as to mix the contents thoroughly. The test-tube receiver should contain sufficient water to allow the end of the retort to dip just below its surface. Now heat the retort gently. The bromine is liberated and distills over (R). Keep the receiver cool by pouring cold water over it. Continue the heating until all the bromine has distilled over. Remove the stopper from the retort before the heat is withdrawn.

Note the properties of the bromine collected in the bottom of the receiver. Has any dissolved in the water? What property is implied in the name of the element? Test the bleaching property of the aqueous solution. How does it compare with chlorine as a bleaching agent?

II. IODINE

Preparation and properties of iodine. Clean the retort used in the preparation of bromine and put into it a mixture of 2 g. of potassium iodide and 4 g. of manganese dioxide. Pour over this mixture 5 cc. of sulphuric acid. Insert the stopper and apply a gentle heat (R). Note the vapor of the iodine in the bulb of the retort; also the grayish-black crystals which are soon deposited in the neck of the retort. What property does the name of the element suggest? Prepare some starch

paste by grinding one or two small pieces of starch with a little cold water and adding this to 200 cc. of boiling water. Stir the solution well and allow it to cool. Half fill two test tubes with the clear liquid. To the first add a few drops of a solution of iodine prepared by shaking a small crystal in water. To the second add a few drops of an aqueous solution of potassium iodide. Note the results. Now add to the second tube a little chlorine water. Does the chlorine water alone change the starch? Explain the results. Dissolve a crystal of iodine in alcohol. What is the solution called?

CARBON

1. Fill a small porcelain crucible with pieces of splints and connect it with the bowl of a clay pipe, using clay to make the connections air tight. Heat strongly and observe the character of the volatile matter escaping through the stem of the pipe. Is it combustible? After all the volatile matter has been expelled, examine the residue. What is it? Will it burn? Repeat the experiment, using small pieces of soft coal in place of the wood. Describe the results.

2. Bring a cold porcelain dish into a small luminous Bunsen flame. Note the deposit. What is this form of carbon called? In what other forms does carbon exist? What properties have all of these forms in common?

3. Put $\frac{1}{4}$ test tube full of boneblack into a small flask and pour over it about 50 cc. of water, to which has been added a few drops of a solution of litmus or indigo. Thoroughly mix the contents of the flask; then heat gently for a few minutes and filter. If the filtrate is not decolorized, repeat, using more boneblack. What is the composition of boneblack? By what other name is it known? What use does this experiment suggest for it?

4. Is carbon an active element at ordinary temperatures? Test it with the common acids. How does the charring of wood preserve it?

5. In a hard-glass test tube heat an intimate mixture of 2 or 3 g. of black copper oxide and an equal bulk of powdered charcoal (R). Pass the evolved gases through a little lime-water in a test tube (R). What remains in the hard-glass test tube? What use does this suggest for charcoal?



CARBON DIOXIDE AND CARBONIC ACID

1. Prove its presence in the air exhaled from the lungs by blowing through limewater (R). Hold a wide-mouthed bottle above a small flame so that the hot gaseous products of combustion will collect in it; then quickly add a few cubic centimeters of limewater, cover the mouth of the bottle with the hand, and shake up the contents. What do the results prove?

2. *Preparation* (usual laboratory method). Put some pieces of marble in your hydrogen generator, cover them with water, and add a little strong hydrochloric acid through the funnel tube. Prove that carbon dioxide is evolved. Fill two or three bottles with the gas by displacement of air. (To test when filled, hold a burning splint in the mouth of the bottle.)

3. Devise an experiment to show whether the gas is heavier or lighter than air. Attempt to pour it from one bottle to another, as you would a liquid, and test for its presence in the second bottle with a burning splint.

4. Half fill a small beaker with limewater and pass carbon dioxide through the liquid (R). Continue until the precipitate which forms at first is dissolved (R). Divide the clear liquid into two parts. To the one add a little clear limewater (R); to the other apply heat until it boils rapidly (R).

5. Give the formula and properties of the acid of which the carbonates are salts. Try the action of hydrochloric acid on a small amount (about 1 g.) of each of the following: sodium carbonate, potassium carbonate, magnesium carbonate (R). How can you detect the presence of carbonates? Is the action of sulphuric and nitric acids on carbonates similar to that of hydrochloric acid (R). Why is carbonic acid so readily liberated from carbonates?

A STUDY OF THE FLAME

1. Prepare some charcoal by heating pieces of splints 3 or 4 cm. in length in the bottom of a test tube. Note and account for the difference between the combustion of the splint and that of the charcoal. What are the conditions necessary for the production of a flame? Light a candle and place it so that the flame is against a black background and is not disturbed by air drafts; then note the different cones in the flame. Test the relative temperatures of different parts of the flame by means of narrow strips of splints. Draw a diagram showing the different parts of the flame. Extinguish the candle flame and hold a lighted splint 2 or 3 cm. from the wick in the little column of smoke. The candle is relighted. What does the experiment prove?

2. What elements constitute the main parts of ordinary fuels? What products form when these elements burn in air or oxygen? Devise simple experiments to show the presence of these products in the gases evolved by the burning candle. Account for the moisture deposited on a lamp chimney when the lamp is first lighted.

3. What is meant by the kindling temperature of gases? Press a piece of wire gauze halfway down on a Bunsen flame. Notice that the flame does not extend above the gauze. Is this due to the absence there of combustible gases? Test for their presence by means of a lighted splint. Turn off the gas, then turn it on and ignite it over a piece of wire gauze held horizontally 4 or 5 cm. above the top of the burner. Note the results and explain. How does the miner's safety lamp prevent explosions? Hold a porcelain dish in a small luminous Bunsen flame. Account for the deposition of carbon (lampblack). Does the non-luminous flame deposit carbon? To what is the luminosity of the flame due?

4. Hold a splint horizontally in the base of the Bunsen flame for two or three seconds and note the results. In the same way



determine the relative temperatures of various parts of the flame. Turn the gas down until the flame is 7 or 8 cm. in height, then

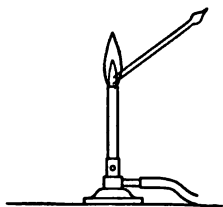


FIG. 27

quickly thrust *vertically* through the center of the flame a piece of white cardboard about 10 cm. in height, the lower end resting against the top of the burner. Remove the cardboard before it is ignited and note from the scorched portions the relative temperatures of different parts of the flame. Draw a diagram to illustrate your results.

That the center of the base of the flame contains the unburned gas may be shown by holding in it the end of an inclined glass tube 12 or 15 cm. in length (Fig. 27), and igniting the gas at the upper end of the tube.

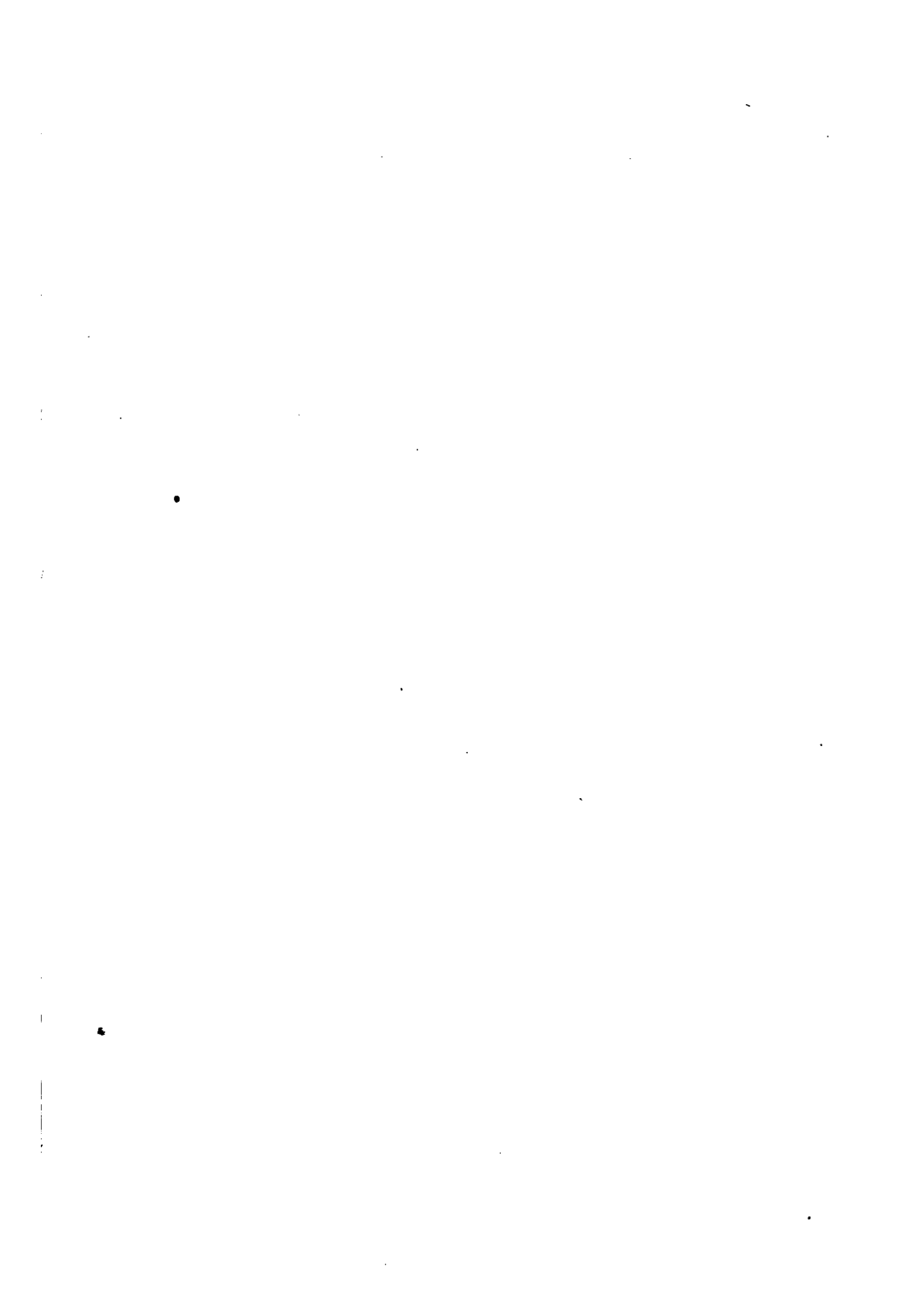
5. *Oxidizing and reducing flames.* Ask the instructor to show you how to produce each of these flames by means of the blowpipe. Then heat in the reducing flame a small amount of an intimate mixture of sodium carbonate and lead oxide (PbO) placed in a small cavity in a piece of charcoal. Have you any evidence of the reduction of the oxide? Try the effect of the oxidizing flame on a small piece of metallic lead on charcoal. Describe the results. Carefully distinguish between an oxidizing flame and a reducing flame. To what does each owe its peculiar property?

DETERMINATION OF EQUIVALENTS

Fit a small wide-mouthed bottle (60 cc.) with a two-holed rubber stopper provided with a funnel tube and a gas delivery tube, as shown in the generator represented in Fig. 15. Weigh out accurately about 1 g. of pure zinc. Without detaching the glass handle, wind your platinum wire tightly around the piece of zinc and carefully transfer it to the generator. Insert the stopper, adjusting the funnel tube so that the end of it comes as near as possible to the bottom of the bottle. Fill a 500-cc. narrow-mouthed bottle with water and invert it in a pneumatic trough, placing the end of the delivery tube just beneath the mouth of the bottle, but not projecting into it. Measure out 2 cc. of sulphuric acid and pour it very slowly on the side of the funnel tube. If care is taken, the acid will drop into the generator without entrapping and carrying with it bubbles of air. Hydrogen is evolved and collects in the 500-cc. bottle. Let the apparatus stand until the zinc is entirely dissolved. This requires about one-half hour. Is the gas in the receiver pure hydrogen? How does its volume compare with that of the hydrogen liberated by the zinc?

It now remains to compute the volume of the gas obtained when measured under standard conditions. Adjust the receiver so that the level of the liquid inside and outside of it is the same; then close the mouth of it firmly with the hand, lift it from the trough, and place it upright on the desk, being careful to allow no water to escape. Now measure the volume of the gas by filling the receiver with water poured from a graduated cylinder. Take the readings of the barometer and thermometer and insert the values below.

Weight of zinc taken	=
Volume of hydrogen liberated	=
Barometric pressure	=
Temperature of hydrogen	=



EXERCISE 28 (continued)

Reduce the volume to standard conditions and calculate the weight of the hydrogen. Compare your answer with the theoretical result. What sources of error are involved in the experiment? Repeat the experiment, using different weights of zinc. Tabulate your results.



I. PHOSPHORUS

Cover the bottom of a wide-mouthed bottle with water to a depth of about 1 cm. Place a piece of phosphorus the size of a pea on a deflagrating spoon and ignite it by touching it with a hot wire. Quickly lower the phosphorus into the bottle and cover its mouth as well as possible with a glass plate. When the phosphorus ceases to burn withdraw the spoon and allow the fumes in the bottle to dissolve in the water. Test the solution with litmus paper. What is present in the water (R)?

II. ARSENIC

1. Note the physical properties of arsenic. Place a bit of the element half as large as a grain of wheat in a cavity on a piece of charcoal and heat it (hood) with a blowpipe (R).

2. Seal the end of a piece of hard-glass tubing about 10 cm. in length and 6 or 7 mm. in diameter. Introduce into it an amount of arsenious oxide equal in bulk to a grain of wheat. Cover this to a depth of 2 or 3 cm. with somewhat finely powdered charcoal which has been previously heated to a high temperature in a porcelain crucible. See that the inner surface of the tube above the charcoal is perfectly clean. Incline the tube and heat the *upper* portion of the charcoal to a high temperature; then, while maintaining the charcoal at this temperature, gradually bring the lower part of the tube also into the flame. The upper part of the tube must be kept as cool as possible. The arsenious oxide is changed into a vapor, which passes over the hot charcoal. Account for the result (R). Cut the tube as near the bottom as possible and remove the charcoal; then, inclining the tube, apply a very *gentle* heat to that portion of it which contains the coating. Note that small white crystals are slowly deposited in the colder portions of the tube (R). Examine these with a magnifying glass. Note their form.



MARSH'S TEST FOR ARSENIC

PRECAUTION:— This experiment must be carried out in a hood and great care taken to prevent the escape of the poisonous arsine into the air of the laboratory.

Arrange an apparatus according to Fig. 16, substituting for the tube *f* a clean, hard-glass tube about 30 cm. long and 8 mm. in diameter, drawn out to a jet at the end. (Use the blast lamp in making the jet.) Generate hydrogen by the usual method, and, after taking the general precautions, ignite it as it escapes from the glass jet. Sufficient acid is added from time to time to cause a gentle evolution of the gas. Now apply a strong heat to the hard-glass tube at a place near its center, using the "wing-top" burner. After a few minutes note whether any deposit forms just beyond the heated portion of the tube. If none forms, the materials are free from arsenic. Now add two drops of a dilute hydrochloric acid solution of arsenious oxide to the generator, rinsing it down the funnel tube with a little water. Continue the heating of the hard-glass tube at the same place. Note the deposit formed on the sides of the tube. Withdraw the heat and hold the lid of a porcelain crucible in the flame. A black deposit of arsenic forms. Test the solubility of this in a solution of sodium hypochlorite. Note the results. Cut the tube containing the deposit so as to just remove the jet and, inclining it, apply a *gentle* heat, as in II, 2, Exercise 29. Account for the results and write the equations for all the reactions involved in the experiment.

EXERCISE 31**I. ANTIMONY**

1. Heat a bit of antimony on charcoal, as in II, 1, Exercise 29. What is formed (R)?

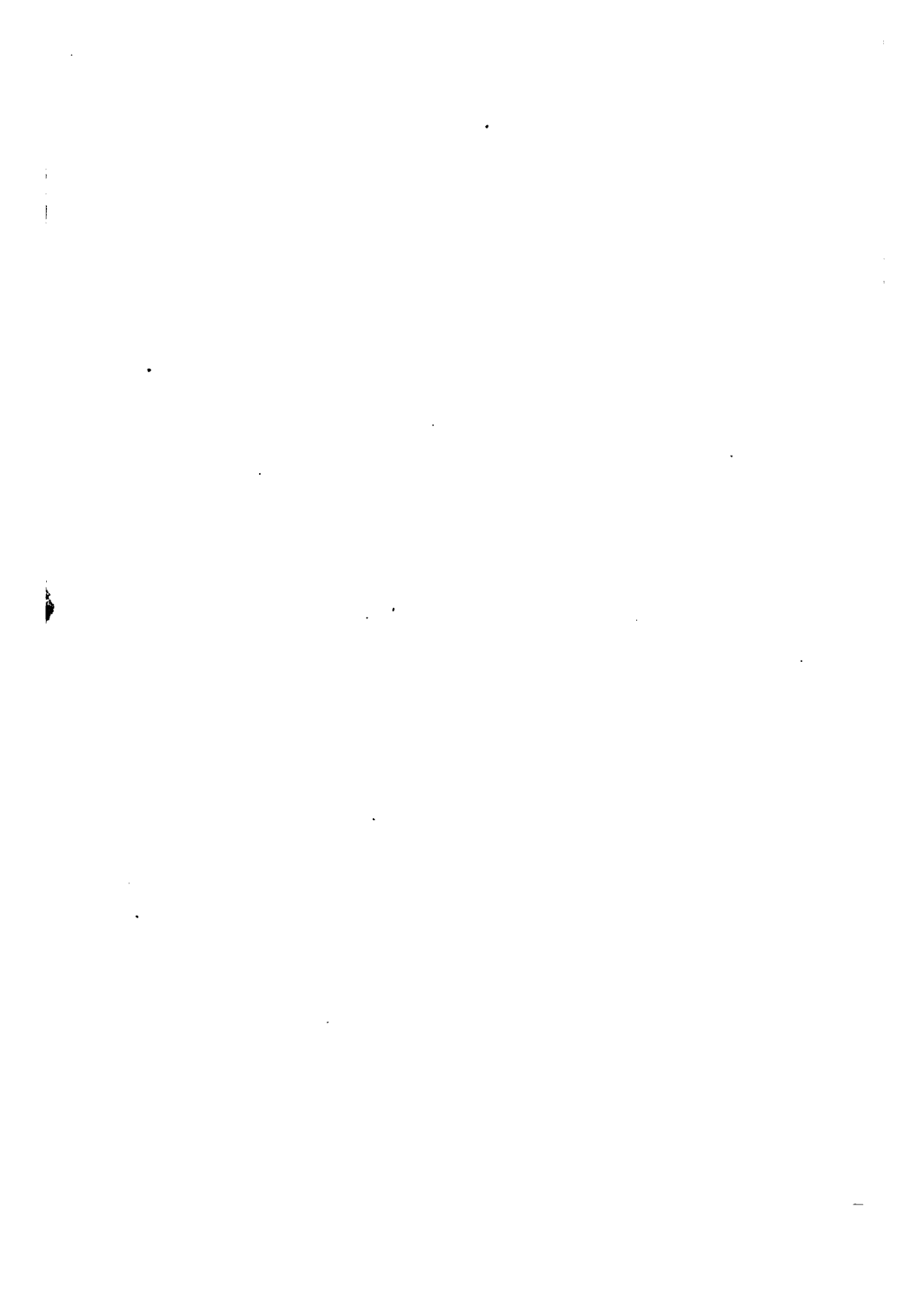
2. Introduce into a test tube a bit of antimony no larger than a grain of wheat, and add about 2 cc. of hydrochloric acid and two or three drops of nitric acid (hood). After the antimony is dissolved, pour the solution into a beaker containing about 100 cc. of water. Explain. Half fill a test tube with the resulting mixture, add hydrochloric acid, a drop at a time, until the solution just clears. Explain. Now pass hydrosulphuric acid through the clear solution until an orange-colored solid forms (R).

3. Boil a small amount of powdered antimony with concentrated nitric acid (hood) until it is changed into a white powder. Dilute the mixture with water, filter, and wash the residue on the filter paper with water. Convince yourself that the residue is not a nitrate. Dissolve a portion of it in sodium hydroxide (R). Dissolve another portion in hydrochloric acid (R).

II. BISMUTH

1. Heat a bit of bismuth on charcoal, as in the case of antimony. What is formed (R)? Contrast the effect of heat on arsenic, antimony, and bismuth.

2. Repeat I, 2, substituting bismuth for antimony and using nitric acid alone as the solvent (R). What color is the precipitate formed by hydrosulphuric acid?



I. BORON

1. Write the names and formulas for three important compounds of boron.

2. Make a little loop on the end of a platinum wire and heat it to redness in a Bunsen flame; then quickly bring the loop in contact with some borax and reheat. The borax adhering to the loop will swell up (why?) and finally form a clear glassy bead. Note the color imparted to the flame. Dip the bead into a dilute solution of a cobalt compound and reheat. Note the results.

3. Dissolve 5 g. of borax in 15 cc. of boiling water. Test the solution with litmus paper. Explain. Add to the hot solution 5 cc. of concentrated hydrochloric acid. Cool the solution and filter off the precipitate (R). Compare the precipitate with borax; first, as to solubility in alcohol, and second, as to the color imparted to a flame.

4. Heat a little boric acid in a porcelain crucible until a clear liquid is formed (R).

II. SILICON

1. Recall the formulas and names of the important acids of silicon.

2. Place 1 cc. of a solution of water glass (Na_2SiO_3) in an evaporating dish, dilute with 10 cc. of water, and add 2 or 3 cc. of hydrochloric acid. Note the gelatinous precipitate (R). Evaporate to dryness and heat the dish gently with the bare flame (R). When cool add water, filter, and examine the residue. What is it?

3. Recall the action of hydrofluoric acid on silica (R).

GENERAL METHODS FOR PREPARING COMPOUNDS

1. Recall the formation of sulphide of iron (Exercise 2) and chloride of copper (Exercise 22). Write the equations for the reactions involved.

2. Place a small bit of copper in an evaporating dish and dissolve (hood) in a few drops of nitric acid. Carefully evaporate the solution. The flame should not touch the dish and should be withdrawn while two or three drops of the liquid still remain in the dish. Note the result (R). Now apply a gentle heat to the residue in the dish (R).

3. Recall the formation of the following compounds and note the principle involved in the preparation of each : hydrochloric acid, nitric acid, ammonia, barium sulphate, sodium sulphate.

4. The following compounds are insoluble: calcium carbonate (CaCO_3), lead sulphate (PbSO_4), barium carbonate (BaCO_3), ferric hydroxide ($\text{Fe}(\text{OH})_3$), silver chloride (AgCl), lead iodide (PbI_2). Prepare a small amount of each in a test tube (R).

5. Place a few cubic centimeters of a solution of a salt of lead in a test tube and suspend in it a thin strip of zinc. Set the tube aside, and after a few minutes note any changes that have taken place. Repeat the experiment, using a solution of a salt of copper and zinc. Discuss the results.

SODIUM

1. Recall experiment I, 1, Exercise 5. Obtain a small piece of sodium. Cut it and note the rapidity with which the freshly cut surface is tarnished. Half fill your evaporating dish with water; then drop the sodium into this and quickly cover the dish with a glass plate (R). After the action has entirely ceased test the liquid with red litmus paper. What compound is dissolved in the water? Convert it into common salt (R). Describe the method.

2. Dissolve 5 g. of sodium carbonate in 20 cc. of water. Prepare common salt from this. How can you be sure that the product contains no unchanged sodium carbonate? Describe the method (R). Treat some of the salt so prepared with sulphuric acid. What gas is evolved (R)?

3. Dissolve 10 g. of ammonium carbonate in 100 cc. of ammonium hydroxide. Saturate this solution with sodium chloride by shaking it with the finely pulverized salt. Pour off the clear solution and pass carbon dioxide through it until a precipitate forms. Filter off the precipitate, dry between filter paper, and examine. (From *Bulletin No. 27*, New York State Education Department.) Write equations for the reactions.

4. Hold a platinum wire in the Bunsen flame until it ceases to impart any color to the flame; then dip it into a solution of a compound of sodium and hold it in the outer film of the base of a Bunsen flame. Note the color. Note the appearance of the sodium flame when viewed through a piece of cobalt glass.



EXERCISE 35**POTASSIUM**

1. Put into a small beaker the theoretical amounts of sodium nitrate and potassium chloride necessary to prepare 10 g. of potassium nitrate. Add 20 cc. of water and boil the mixture gently (R). The sodium chloride formed, being practically as insoluble in hot water as in cold, soon begins to separate. The solution should then be stirred and the heating continued until the volume of the liquid is reduced about one half. Quickly filter the hot solution and set the filtrate aside until cold. Dry the crystals deposited in the filtrate by pressing them between filter papers. Finally recrystallize them from as little hot water as possible. Prove the composition of the purified crystals. What weights of sodium nitrate and potassium chloride did you take?

2. Repeat 4, Exercise 34, using a solution of any compound of potassium. Note the color of the flame. Repeat, using a solution containing compounds of both sodium and potassium. How could you detect both sodium and potassium when present in the same solution?

3. Test with litmus paper solutions of the following compounds: sodium carbonate, sodium hydrogen carbonate, potassium carbonate. Account for the results.



AMMONIUM COMPOUNDS

1. Prepare a small amount of ammonium chloride by neutralizing a solution of ammonium hydroxide with hydrochloric acid and evaporating to dryness on a water bath. Treat the resulting product with sodium hydroxide as in I, 1, Exercise 16. Note the result. This reaction serves as a good test for ammonium compounds.

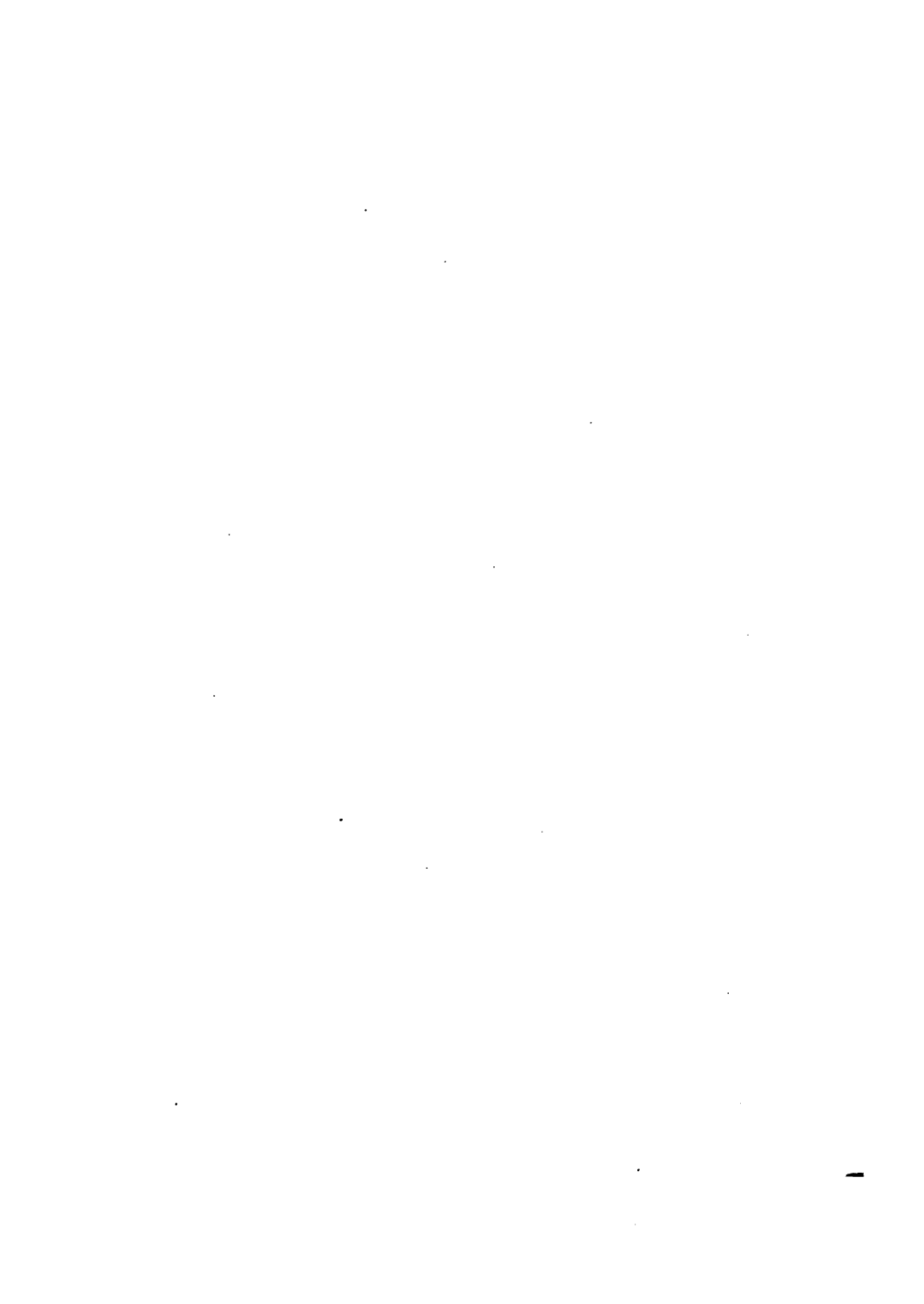
2. Pour 2 or 3 cc. of a solution of ferrous sulphate into each of two test tubes. To the one add a few drops of an aqueous solution of hydrosulphuric acid; to the other a few drops of a solution of ammonium sulphide. Account for the results. When is ammonium sulphide used in place of hydrosulphuric acid? How is it prepared? What changes does it undergo on standing?

3. Prepare a dilute solution of ammonium carbonate and add a few drops of it to solutions of compounds of each of the following elements: barium, lead, iron, zinc, manganese. Explain. What use does this suggest for ammonium carbonate?

4. Test compounds of lithium, sodium, and potassium with the spectroscope.

DETECTION OF COMPOUNDS OF THE ALKALI METALS

Ask the instructor for unknown compounds of sodium, potassium, or ammonium, and see if you can identify them. Recall such reactions of carbonates, sulphates, nitrates, sulphites, sulphides, chlorides, bromides, and iodides as will serve to identify them.



CALCIUM

1. Dissolve 2 or 3 g. of marble in hydrochloric acid (R). What does the effervescence indicate? Evaporate the solution to dryness. What is the composition of the residue? Expose a small piece of it to the air for an hour and account for the results. Dissolve the remainder in a little water and divide into two portions. To the one add a few drops of ammonium carbonate (R); to the other add a few drops of solutions of ammonium hydroxide and disodium phosphate (R).

2. Place some small pieces of marble in a porcelain crucible and apply a strong heat for about fifteen minutes. The crucible must not be covered and should be tipped slightly. When cool, drop the residue into 25 cc. of water and stir. Then filter and divide the filtrate into two parts. Blow exhaled air through one portion, and add one or two drops of ferric chloride to the other portion. Explain all reactions.

3. Heat a crystal of gypsum in a test tube (R). Place on a glass plate a button which has been smeared with a drop of oil. Pour over the button a thick paste prepared by adding water to plaster of Paris. Set it aside until it hardens; then remove the button and note the result. What causes the paste to harden? For what is plaster of Paris used?

4. Shake 1 g. of calcium sulphate with 10 cc. of water in a test tube, filter, and test the filtrate for the presence of sulphates. Is calcium sulphate soluble in water? Recall the conditions under which calcium carbonate is soluble in water. Give two methods for removing the calcium carbonate from such a solution.

BLEACHING POWDER

Arrange an apparatus according to Fig. 28. *A* represents a 250-cc. flask in which chlorine is generated according to II, 3, Exercise 22, using 15 g. of manganese dioxide and 75 cc. of hydrochloric acid. The chlorine must be evolved *slowly*, for

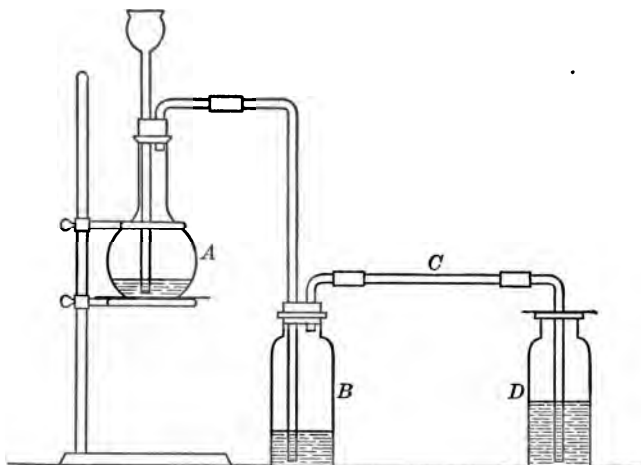
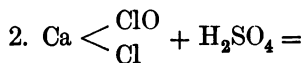
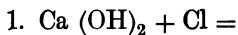


FIG. 28

which reason only a *very gentle* heat is applied to the flask. The gas is conducted through a little water in *B*, and then into the tube *C*, which is half filled with slaked lime. The bottle *D* contains a solution of sodium hydroxide. When all of the chlorine has passed over from *A*, disconnect the apparatus and transfer the contents of the tube *C* to a beaker (200 cc.–300 cc.) and pour over it a little sulphuric acid diluted with an equal volume of water. Hang a moist strip of red calico in the beaker and cover with a glass plate. Complete the following equations:





I. MAGNESIUM

1. Recall the combustion of magnesium (R). Convert 2 or 3 g. of magnesium carbonate into the chloride. Evaporate the solution to complete dryness, heating the residue gently with the bare flame. What is the composition of this residue? Are waters containing magnesium chloride objectionable for use in steam boilers? Give reason for your answer.

2. Add a few drops of ammonium hydroxide to a solution of magnesium sulphate (R). Repeat, first diluting the magnesium sulphate with an equal volume of ammonium chloride solution. Now add to this a solution of disodium phosphate (R). The precipitate has the composition MgNH_4PO_4 .

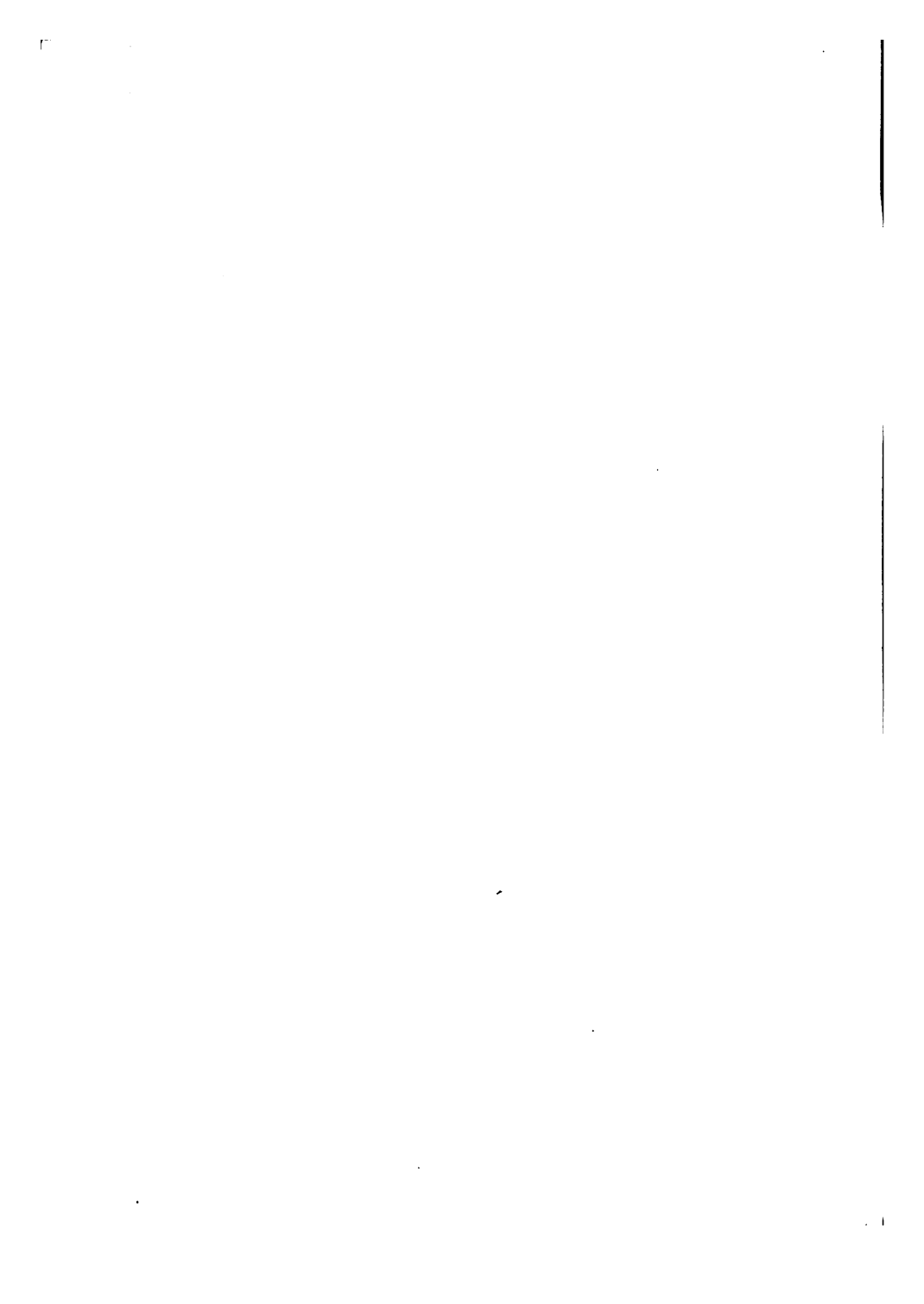
II. ZINC

1. Place a small piece of zinc on charcoal and heat it in the oxidizing flame of the blowpipe (R). Note the color of the film deposited. Is the color of the film affected by heat?

2. The following compounds of zinc are insoluble: hydroxide, sulphide, carbonate, and phosphate. Prepare small amounts of each. Give the color of each and write equations for all reactions involved in their preparation. (Disodium phosphate precipitates the normal zinc phosphate.)

III. CADMIUM

Examine the physical properties of cadmium. Prepare the insoluble sulphide from a solution of the chloride (R). Note the color of the sulphide.



ALUMINIUM

1. Note the physical properties of aluminium. Dissolve a small piece in hydrochloric acid (R). Dilute the solution to about 10 cc. and divide into two equal parts. To the first add ammonium hydroxide until the solution reacts alkaline (R); to the second add sodium hydroxide, a drop at a time, until a precipitate forms (R); then add a few drops more and note the result (R).

2. Prepare some aluminium hydroxide and heat it on charcoal in the blowpipe flame (R). Moisten the residue with a drop or two of a solution of cobalt nitrate and reheat. Note the result. Advantage is taken of this property in detecting the presence of aluminium.

3. Add a few drops of a solution of sodium carbonate to a solution of any salt of aluminium (R). Filter off the precipitate and wash it with water. Make appropriate tests to determine whether it is a carbonate. Repeat the experiments, substituting ammonium sulphide for sodium carbonate (R).

4. Mix a little dry, powdered alum and bicarbonate of soda. Is there any visible reaction? Now pour a little water over the mixture and note the result. Explain.

5. Add 1 or 2 cc. of ammonium hydroxide to some water colored with a few drops of litmus solution. Is the litmus precipitated? Add a similar amount of ammonium hydroxide to 5 or 10 cc. of a dilute solution of alum (R). Now add to the resulting mixture two or three drops of litmus solution, shake the mixture thoroughly, and set it aside until the precipitate settles. Note the result and explain.

6. Saturate a piece of cotton cloth with a solution of alum; then dip it into a solution of ammonium hydroxide. Wring out the cloth and boil it in a concentrated solution of litmus. Repeat with a piece of cloth which has not been treated with the solutions of alum and ammonium hydroxide. Explain the results.



I. IRON

1. Place 5 g. of iron (wire or tacks) in a beaker and pour over it 15 cc. of water. Now add 4 cc. of concentrated sulphuric acid and heat very gently (hood) until a vigorous evolution of gas takes place (R); then cover the beaker with a watch glass and set it aside in the hood until near the end of the laboratory period. Add 10 cc. of water and heat slowly until the liquid boils, stirring the mixture constantly. Filter off any undissolved solids, collecting the filtrate in an evaporating dish. Set the filtrate in your desk until the next laboratory period; then examine the crystals and save some of them for Exercise 46.

2. Dissolve a small amount of iron in dilute hydrochloric acid (R). Filter off any residue. Divide the solution into two parts. To the one add ammonium hydroxide until strongly alkaline (R). Filter. Explain the change in the color of the precipitate on exposure to the air (R). To the second part add two or three drops of nitric acid and boil for a few minutes (R). Explain the effect of the nitric acid on the color of the solution. Now cool the solution and add ammonium hydroxide (R). Account for the difference in the action of ammonium hydroxide on the two solutions.

3. Dissolve 10 g. of crystals of ferrous sulphate in 20 cc. of water. Add to the solution the calculated amount of sulphuric acid necessary to convert the salt to the ferric state; heat to boiling and add nitric acid, a drop at a time, until the color no longer changes. Evaporate (hood) to a sirupy mass and dissolve in a little hot water. Add to the resulting solution the calculated amount of ammonium sulphate necessary to form ferric ammonium sulphate, and heat gently until the salt is dissolved. Set the resulting solution aside until the next laboratory period; then examine the form of the crystals. Write the equations for the reactions involved.

II. COBALT AND NICKEL

Test separate solutions of a salt of cobalt and nickel with the borax bead; with a solution of sodium hydroxide; with ammonium sulphide. Note the results.

I. COPPER

1. Recall the action of sulphuric and nitric acids on copper (R); also of sulphur and chlorine on copper (R). Insert a nail into a solution of copper sulphate. Account for the result (R).

2. To a cold solution of copper sulphate add one half its volume of sodium hydroxide solution (R). Now heat to boiling and account for the change in the color of the precipitate.

3. Try the action of aqueous solutions of hydrosulphuric acid and ammonium sulphide on copper sulphate (R). Add one drop of ammonium hydroxide to a dilute solution of copper sulphate (R); now continue to add the ammonium hydroxide, drop by drop, until the precipitate is dissolved. How does the color of this solution compare with that of the original solution?

II. MERCURY

1. Note the physical properties of mercury. Place a globule of it (as large as a grain of wheat) in a small beaker and add (hood) just enough strong nitric acid to dissolve it. Write the equation for the reaction on the supposition that mercuric nitrate is formed and nitric oxide evolved. Dilute the product with 10 cc. of water and place a copper penny in the solution. After a few minutes remove the coin and polish it with a piece of cloth. Account for the result.

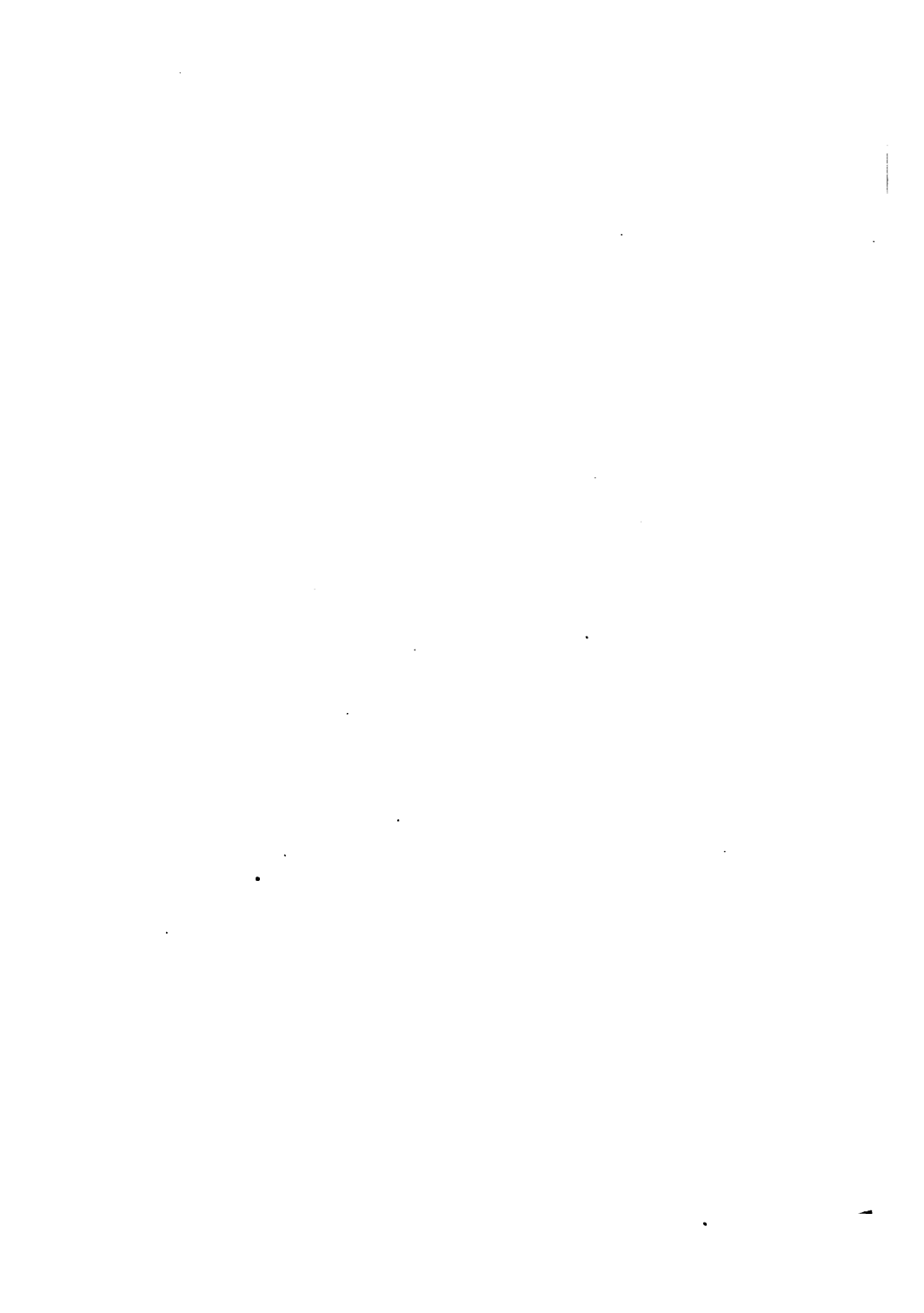
2. For what purpose have we used mercuric oxide? Place $\frac{1}{2}$ g. of it in a test tube and dissolve it in as little nitric acid as possible (R). Then add water until the test tube is one fourth full. Into a second test tube pour a similar volume of a solution of mercurous nitrate. Now add a little hydrochloric acid to each test tube. What conclusions do you draw in reference to the solubility of the two chlorides of mercury? Write the formulas and names (chemical and common) of each.



EXERCISE 44**SILVER**

1. Place a silver dime in a small beaker and add (hood) sufficient nitric acid to dissolve it. The solution may be hastened by applying a gentle heat. When the solution is complete dilute the product with about 25 cc. of water. Account for the color of the liquid. Now add a solution of sodium chloride until a precipitate ceases to form. On stirring, the precipitate (what is it ?) settles to the bottom of the beaker. Carefully decant the clear, supernatant liquid and test it for the presence of copper (I, 3, Exercise 43). Wash the precipitate two or three times by pouring hot water over it and decanting. Finally remove any remaining water by filtration. Mix the product with an equal bulk of sodium carbonate, transfer to a small cavity in a piece of charcoal, and heat with a blowpipe. The silver salt is gradually reduced to metallic silver, which will fuse into a globule if sufficient heat is applied.

2. Prepare small amounts of the chloride, bromide, and iodide of silver (R). Expose the test tubes containing the precipitates to the sunlight and note any changes. For what are these compounds used ? Prepare sufficient silver sulphide to note its color.



I. LEAD

1. Note the physical properties of the metal. Heat a small piece on charcoal in the oxidizing flame. Note the coating formed (R).

2. Place 1 g. of the metal in an evaporating dish (hood) and add 20 cc. of water and 5 cc. of nitric acid. Support the dish on a wire gauze and heat *gently* until the metal is dissolved (R). Evaporate until the volume is about one half of the original. What is the composition of the white body which separates? Dilute to 100 cc. and filter, if necessary, to obtain a clear solution. Now test small portions of this with hydrosulphuric acid, sulphuric acid, potassium chromate, and ammonium carbonate, respectively (R). Note the color and composition of the precipitates. Add a few drops of hydrochloric acid to a test tube one fourth full of the solution (R). Heat this to boiling, and if the solution does not become clear, add just enough boiling water to dissolve the precipitate; then set it aside until cool and note the result. How can you distinguish between lead chloride and silver chloride? In the remainder of the solution, formed by dissolving the lead in nitric acid, suspend a piece of zinc. Set aside for half an hour and note the result (R). Test the solution for the presence of zinc, giving the equations for method used.

II. TIN

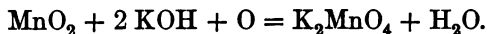
1. Note the physical properties of tin. Dissolve a small piece in concentrated hydrochloric acid (R). Cool, dilute with a little water, and add gradually to a few drops of mercuric chloride (R). Note and explain any change in the color of the precipitate.

2. Dissolve a small piece of tin in aqua regia. What compound is formed? Pour a few drops of the solution into an aqueous solution of hydrosulphuric acid (R).



MANGANESE

1. *Manganates and permanganates.* Grind in a mortar 5 g. each of manganese dioxide and potassium hydroxide and 2.5 g. of potassium chlorate. Transfer the mixture to a small iron dish and heat to redness until the mass fuses and becomes green. The reaction expressed by the following equation takes place:



What is the composition of the green substance? When the mass is cool add water. After a few minutes decant the solution from any residue. Now pass carbon dioxide through the solution. What evidence have you of any change? The reaction is expressed by the equation



Dissolve a small crystal of ferrous sulphate, prepared in I, 1, Exercise 42, in a little water and add two or three drops of sulphuric acid. Now add the solution of potassium permanganate, drop by drop, until the color remains permanent. Write the equations for the reaction (the manganese and potassium are both changed to sulphates).

2. Add some hydrochloric acid to a few crystals of potassium permanganate and account for the results (R).

3. *Salts of manganese.* In the above compounds manganese acts as an acid-forming element. It also acts as a base-forming element. Try the action of ammonium sulphide, ammonium carbonate, and sodium hydroxide, respectively, on a solution of manganese chloride (R).

CHROMIUM

1. *Chromates and dichromates.* Write the formula for potassium chromate; for potassium dichromate. Is the chromium an acid-forming or base-forming element in these compounds? Add two or three drops of nitric acid to a little potassium chromate solution. Note the result (R). To the resulting solution add a few drops of potassium hydroxide solution. Explain (R).

2. Try the effect of a solution of potassium chromate on a solution of a compound of lead (R); also on a solution of a compound of barium (R).

3. Add a few drops of hydrochloric acid to a little solid potassium chromate and explain the results (R). Repeat, using potassium dichromate (R).

4. *Salts of chromium.* Prepare a solution of a salt of chromium and treat portions of it with the following reagents: ammonium sulphide, sodium carbonate, sodium hydroxide. Write all the equations.



FERMENTATION

Prepare an apparatus according to Fig. 29. About 100 g. of commercial glucose are dissolved in 1 l. of water and the solution is poured into flask *A*. This flask is connected with the bottle *B*, which is partially filled with limewater. Sufficient kerosene is added to *B* to form a layer on the limewater about

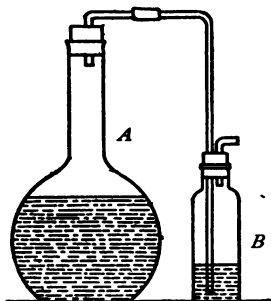


FIG. 29

$\frac{1}{2}$ cm. in depth. About one half of a cake of compressed yeast is cut into pieces and introduced into flask *A*, and the apparatus is connected, as shown in the figure. Set the apparatus aside for several hours in a warm place. The reaction takes place best at a temperature of from 25 to 30 degrees. Bubbles of gas will be seen to escape through the limewater in *B* and a precipitate of calcium carbonate forms. The kerosene on the limewater protects it from the action of the carbon dioxide of the atmosphere. After the fermentation has ceased, distill off 2 or 3 cc. of the liquid from *A* and test it with a flame. Describe the results and write the equations for the reactions involved.

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SAPONIFICATION

Add 10 cc. of alcohol to 4 or 5 g. of lard in an evaporating dish. To the resulting mixture add about 1 cc. of a 50 per cent solution of sodium hydroxide in water. Evaporate carefully, stirring the mixture constantly until the odor of alcohol can no longer be detected. Write the equation for the reaction on the supposition that the lard is composed of palmitin. What remains in the dish? Add a few cubic centimeters of cold water, stir well, and filter, if necessary, to obtain a clear solution. Add a little of the resulting solution to test tubes containing calcium sulphate and magnesium sulphate, respectively. Explain the results.



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